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(54)【発明の名称】発光素子の製造方法

(57)【要約】
【課題】高輝度、発光効率、耐久性に優れた発光素子の製造方法を提供する。
【解決手段】発光層を含む少なくとも一層の有機層を有する発光素子であり、有機層に含まれる化合物と、少なくとも一つの精製処理により精製された少なくとも一種の溶剤とを含む塗布液を用いて、少なくとも一層の有機層を湿式製膜により形成する発光素子の製造方法。
【選択図】 なし

【特許請求の範囲】
【請求項１】
発光層を含む少なくとも一層の有機層を有する発光素子であり、有機層に含まれる化合物と、少なくとも一つの精製処理により精製された少なくとも一種の溶剤とを含む塗布液を用いて、少なくとも一層の有機層を湿式製膜により形成する発光素子の製造方法。
【請求項２】
少なくとも一つの精製処理が、脱水分処理、及びカラム精製の少なくとも一つである請求項１に記載の製造方法。
【請求項３】
少なくとも一つの精製処理が、脱水分処理、かつカラム精製である請求項２に記載の製造方法。
【請求項４】
精製された少なくとも一種の溶剤の含水量が１００ｐｐｍ以下である請求項１乃至３のいずれかに記載の製造方法。

【請求項５】
精製された少なくとも一種の溶剤が少なくとも一種のハロゲン系溶剤である請求項１乃至４のいずれかに記載の製造方法。
【請求項６】
塗布液が燐光発光材料を含む請求項１乃至５のいずれかに記載の製造方法。

【発明の詳細な説明】
【０００１】
【発明の属する技術分野】
本発明は、バックライト、フラットパネルディスプレイ、照明光源、表示素子、電子写真、有機半導体レーザー、記録光源、露光光源、読み取り光源、標識、看板、光通信デバイス等の分野に利用可能な発光素子（有機電界発光素子）の製造方法に関する。
【０００２】
【従来の技術】
湿式製膜法で形成される発光素子は、三重項励起子を利用する発光素子（燐光発光素子）、一重項励起子を利用する発光素子（蛍光発光素子）の各々の発光形態において、蒸着型素子に比べ発光効率、耐久性共に劣る。湿式製膜法で形成される発光素子は、安価で大面積が可能という大きなメリットがある。発光効率、耐久性の優れた湿式製膜法による得られる発光素子が望まれる。
【０００３】
発光素子の分野において、蒸着型素子での製造では、有機化合物は昇華精製されるために精製を行う必要がなかった。特開２００１－２１４１５９では、発熱による劣化を抑制するを目的として、発光性有機化合物に複数回の精製処理を施すことにより、該発光性有機化合物からなる薄膜のナトリウム、又はカリウムのイオン性不純物濃度を０．０１ｐｐｍ以下にすることが開示されている。
【０００４】
【発明が解決しようとする課題】
本発明は、前記諸問題を解決し、発光効率及び発光輝度に優れ、耐久性に優れた発光素子の製造方法を提供することを目的とする。
【０００５】
【課題を解決するための手段】
本発明者は、有機化合物の精製処理ではなく、本発明では湿式製膜法による発光素子の製造方法における塗布液に問題を有することを見つけ、前記課題を解決するための手段を見出した。即ち、
１．発光層を含む少なくとも一層の有機層を有する発光素子であり、有機層に含まれる化合物と、少なくとも一つの精製処理により精製された少なくとも一種の溶剤とを含む塗布液を用いて、少なくとも一層の有機層を湿式製膜により形成する発光素子の製造方法。

2．少なくとも一つの精製処理が、脱水分処理、及びカラム精製の少なくとも一つである前記１に記載の製造方法。

3．少なくとも一つの精製処理が、脱水分処理、かつカラム精製である前記２に記載の製造方法。

4．精製された少なくとも一種の溶剤の含水量が１００ｐｐｍ以下である前記１乃至３のいずれかに記載の製造方法。

5．精製された少なくとも一種の溶剤が少なくとも一種のハロゲン系溶剤である前記１乃至４のいずれかに記載の製造方法。

6．塗布液が燐光発光材料を含む前記１乃至５のいずれかに記載の製造方法。

【０００６】

【発明の実施の形態】

本発明は、発光層を含む少なくとも一層の有機層を有する発光素子であり、有機層に含まれる化合物と、精製処理された少なくとも一種の溶剤とを含む塗布液を用いて、少なくとも一層の有機層を湿式製膜により形成することを特徴とする発光素子の製造方法である。

【０００７】

本発明では、精製処理は、特に限定されず、例えば、脱水分処理、カラム法、蒸留法、冷却再結晶法等が採用できる。少なくとも一つの精製処理が、脱水分処理、及びカラム精製の少なくとも一つを用いることが好ましく、少なくとも一つの精製処理が、脱水分処理かつカラム精製（脱水分処理とカラム精製との併用）であることがより好ましい。

【０００８】

本発明では、塗布液を調液する前に溶剤の精製処理を行うことが好ましい。前とは調液前１週間以内であり、好ましくは３日以内、更に好ましくは１日以内である。これよりも前に精製を行うと精製の効果が少なくなる。溶剤の精製処理により、発光素子の発光効率、耐久性などを大幅に改良できる。

【０００９】

塗布液の溶剤は、溶解性の点から少なくとも一種のハロゲン系溶剤を用いることが好ましい。中でも塩素系溶剤が好ましく、例えば四塩化炭素、クロロホルム、ジクロロメタン、１，２－ジクロロエタン、１，１－ジクロロエタン、クロロベンゼン、ジクロロベンゼン等を挙げることができる。

【００１０】

上記溶剤以外に、下記に列挙する他の溶剤と混合して用いることもできる。他の溶剤としては、アセトン、メチルエチルケトン、シクロヘキサノン等のケトン系溶剤、メタノール、エタノール、１－プロパノール、２－プロパノール、ブタノール等のアルコール系溶剤、ベンゼン、トルエン、キシレン等の芳香族炭化水素系溶剤、酢酸メチル、酢酸エチル等のエステル系溶剤、ジエチルエーテル、テトラヒドロフラン、ジオキサン等のエーテル系溶剤、ジメチルアセトアミド、ジメチルホルムアミド等のアミド系溶剤、ジメチルスルホキシド等が挙げられる。

【００１１】

塗布液は、後に詳述するが、少なくとも一種の有機層に用いられる成分（有機化合物など）と、それを溶解もしくは分散するための少なくとも一種の溶剤とを含み、適宜、発光性能や塗布を最適化するために他の成分を含んでもよい。

【００１２】

溶剤の脱水分処理の方法は、特に限定されることはなく、例えば、乾燥剤を溶剤中に入れ放置し、濾過後蒸留により脱水処理を行うことができる。

【００１３】

乾燥剤は、特に限定されることは無いが、無水硫酸ナトリウム、無水硫酸カルシウム、硫酸マグネシウム、硫酸ストロンチウム、硫酸バリウム、酸化バリウム、酸化カルシウム、酸化マグネシウム、モレキュラーシーブ、ゼオライト等を好適に選ぶことができる。乾燥剤により乾燥後、濾過して使用することができるが、さらに蒸留して使用することが好ましい。乾燥後は水分量１００ｐｐｍ以下の不活性ガス雰囲気下で保存することが好ましい

【００１４】
脱水分処理及び／又はカラム精製により精製処理された少なくとも一種の溶剤の含水量は、１００ｐｐｍ以下が好ましく、５０ｐｐｍ以下がより好ましく、３０ｐｐｍ以下がさらに好ましい。溶剤の脱水分処理及び／又はカラム精製により、水分及び／又は発光素子の発光性能を劣化させる不純物を削減できる。
【００１５】
カラム精製では、カラムに用いる充填剤は特に限定されず、例えば、シリカゲル、アルミナ、カチオン性イオン交換樹脂、アニオン性イオン交換樹脂等を好適に使用できる。また塩基性、中性、及び酸性のいずれの充填剤も使用できる。
【００１６】
充填剤の量は精製する溶剤１００ｍｌに対して、５０ｇ以上１ｋｇ以下であることが好ましい。これよりも少ないと精製の効果は小さくなり、これよりも多いと充填剤の量が無駄になり生産効率が低下する。また精製時の流量は１ｍｌ／分以上１０００ｍｌ／分以下であることが好ましい。これよりも流量が少ないと生産効率が悪くなる。またこれよりも多いと精製の効果は小さく好ましくない。
【００１７】
塩素系溶剤をカラム精製することにより、ラジカルやカチオン等の塩素活性種が除かれると推測される。特に塩素活性種が溶剤中に残留すると、後述する有機層に含まれる材料を塩素化し、耐久性悪化、効率悪化の原因となる。
【００１８】
本発明の製造方法において特に好ましい態様は、少なくとも一つの精製処理された少なくとも一種の溶剤と燐光発光材料とを含有する塗布液を用いた発光層の形成である。精製処理はカラム精製が好ましく、カラム精製と脱水分処理との併用がより好ましい。燐光発光性材料は、下記に詳述するように、好ましくはオルトメタル化金属錯体などが挙げられる。溶剤は少なくとも一種のハロゲン系溶剤を含む溶剤であることが好ましい。
【００１９】
特に好ましい態様における精製処理された溶剤は、ある濃度以下にラジカルやカチオン等の塩素活性種などの不純物を除去した溶剤であることが好ましい。発光層において塩素化されたオルトメタル化金属錯体の濃度はある濃度以下であることが好ましい。
【００２０】
（発光素子）
以下に本発明における発光素子について詳細に述べる。本発明の有機層は、発光素子に用いることのできる層を意味し、有機層は主として有機化合物からなり、具体的には、発光性有機層、電子輸送性有機層、ホール輸送性有機層、電子注入層、ホール注入層等として作用効果を有する化合物を含有する層である。なお、以下において「誘導体」という用語はその化合物自身、及びその誘導体を意味し、例えばクマリン誘導体はクマリン及びその誘導体を意味する。
【００２１】
－－有機層の構成－－
前記有機層の前記発光素子における形成位置としては、特に制限はなく、発光素子の用途や目的に応じて適宜選択することができるが、第一の電極、及び第二の電極の間に形成されるのが好ましい。前記第一の電極、及び第二の電極は、透明電極、又は背面電極のどちらでもよい。有機層は、前記透明電極又は前記背面電極上の前面又は一面に形成される。前記有機層の形状、大きさ、厚み等については、特に制限はなく、目的に応じて適宜選択することができる。
【００２２】
具体的な層構成としては、透明陽極／発光層／陰極、透明陽極／発光層／電子輸送層／陰極、透明陽極／正孔輸送層／発光層／電子輸送層／陰極、透明陽極／正孔輸送層／発光層／陰極、透明陽極／発光層／電子輸送層／電子注入層／陰極、透明陽極／正孔注入層／正

孔輸送層／発光層／電子輸送層／電子注入層／陰極等が挙げられる。

【００２３】

－－発光層－－

本発明に用いられる発光層は、少なくとも一種の発光材からなり、必要に応じて正孔輸送材、電子輸送材、ホスト材を含んでも良い。　本発明に用いられる発光材としては特に限定されることはなく、蛍光発光性化合物又は燐光発光性化合物であれば用いることができる。

【００２４】

例えば蛍光発光性化合物としては、ベンゾオキサゾール誘導体、ベンゾイミダゾール誘導体、ベンゾチアゾール誘導体、スチリルベンゼン誘導体、ポリフェニル誘導体、ジフェニルブタジエン誘導体、テトラフェニルブタジエン誘導体、ナフタルイミド誘導体、クマリン誘導体、ペリレン誘導体、ペリノン誘導体、オキサジアゾール誘導体、アルダジン誘導体、ピラリジン誘導体、シクロペンタジエン誘導体、ビススチリルアントラセン誘導体、キナクリドン誘導体、ピロロピリジン誘導体、チアジアゾロピリジン誘導体、スチリルアミン誘導体、芳香族ジメチリデン化合物、８－キノリノール誘導体の金属錯体や希土類錯体に代表される各種金属錯体、ポリチオフェン誘導体、ポリフェニレン誘導体、ポリフェニレンビニレン誘導体、ポリフルオレン誘導体等の高分子化合物等が挙げられる。これらは一種もしくは二種以上を混合して用いることができる。

【００２５】

燐光発光性化合物としては特に限定されることはないが、オルトメタル化金属錯体、又はポルフィリン金属錯体が好ましい。

【００２６】

前記オルトメタル化金属錯体とは、例えば山本明夫著「有機金属化学－基礎と応用－」１５０頁、２３２頁、裳華房社（１９８２年発行）やH. Ｙｅｒｓｉｎ著「Ｐｈｏｔｏｃｈｅｍｉｓｔｒｙ　ａｎｄ　Ｐｈｏｔｏｐｈｉｓｉｃｓ　ｏｆ　Ｃｏｏｄｉｎａｔｉｏｎ　Ｃｏｍｐｏｕｎｄｓ」７１～７７頁、１３５～１４６頁、Ｓｐｒｉｎｇｅｒ－Ｖｅｒｌａｇ社（１９８７年発行）等に記載されている化合物群の総称である。該オルトメタル化金属錯体を含む前記有機層は、高輝度で発光効率に優れる点で有利である。

【００２７】

オルトメタル化金属錯体を形成する配位子としては、種々のものがあり、上記文献にも記載されているが、その中でも好ましい配位子としては、２－フェニルピリジン誘導体、７，８－ベンゾキノリン誘導体、２－（２－チエニル）ピリジン誘導体、２－（１－ナフチル）ピリジン誘導体、２－フェニルキノリン誘導体等が挙げられる。これらの誘導体は必要に応じて置換基を有しても良い。オルトメタル化金属錯体は、前記配位子のほかに、他の配位子を有していてもよい。

【００２８】

本発明で用いるオルトメタル化金属錯体は　Ｉｎｏｒｇ．Ｃｈｅｍ．　１９９１年，　３０号，　１６８５頁．　，同　１９８８年，　２７号，　３４６４頁．　，同　１９９４年，　３３号，　５４５頁．　Ｉｎｏｒｇ．Ｃｈｉｍ．Ａｃｔａ　１９９１年，１８１号，　２４５頁．　Ｊ．Ｏｒｇａｎｏｍｅｔ．Ｃｈｅｍ．　１９８７年，　３３５号，　２９３頁．Ｊ．Ａｍ．Ｃｈｅｍ．Ｓｏｃ．　１９８５年，　１０７号，　１４３１頁．　等、種々の公知の手法で合成することができる。

【００２９】

前記オルトメタル化錯体の中でも、三重項励起子から発光する化合物が本発明においては発光効率向上の観点から好適に使用することができる。また、ポルフィリン金属錯体の中ではポルフィリン白金錯体が好ましい。

【００３０】

前記燐光発光性の化合物は１種単独で使用してもよいし、２種以上を併用してもよい。また、前記蛍光発光性化合物と燐光発光性化合物を同時に用いても良い。本発明においては、発光輝度、発光効率の点から、前記燐光発光性化合物を用いることが好ましい。

【００３１】
前記燐光発光性の化合物の前記発光層における含有量としては、特に制限はなく、目的に応じて適宜選択することができ、例えば、０．１～７０重量％であり、１～２０重量％が好ましい。前記燐光発光性の化合物の含有量が０．１～７０重量％でない場合にはその含有効果が十分に発揮されないことがあり、１～２０重量％であるとその含有効果が十分である。
【００３２】
前記ホスト化合物とは、その励起状態から前記燐光発光性の化合物へエネルギー移動が起こり、その結果、該燐光発光性の化合物を発光させる機能を有する化合物のことである。
【００３３】
前記ホスト材としては励起子エネルギーを発光材にエネルギー移動できる化合物ならば特に制限はなく、目的に応じて適宜選択することができ、具体的にはカルバゾール誘導体、トリアゾール誘導体、オキサゾール誘導体、オキサジアゾール誘導体、イミダゾール誘導体、ポリアリールアルカン誘導体、ピラゾリン誘導体、ピラゾロン誘導体、フェニレンジアミン誘導体、アリールアミン誘導体、アミノ置換カルコン誘導体、スチリルアントラセン誘導体、フルオレノン誘導体、ヒドラゾン誘導体、スチルベン誘導体、シラザン誘導体、芳香族第三アミン化合物、スチリルアミン化合物、芳香族ジメチリデン系化合物、ポルフィリン系化合物、アントラキノジメタン誘導体、アントロン誘導体、ジフェニルキノン誘導体、チオピランジオキシド誘導体、カルボジイミド誘導体、フルオレニリデンメタン誘導体、ジスチリルピラジン誘導体、ナフタレンペリレン等の複素環テトラカルボン酸無水物、フタロシアニン誘導体、８－キノリノール誘導体の金属錯体やメタルフタロシアニン、ベンゾオキサゾールやベンゾチアゾールを配位子とする金属錯体に代表される各種金属錯体ポリシラン系化合物、ポリ（Ｎ－ビニルカルバゾール）誘導体、アニリン系共重合体、チオフェンオリゴマー、ポリチオフェン等の導電性高分子オリゴマー、ポリチオフェン誘導体、ポリフェニレン誘導体、ポリフェニレンビニレン誘導体、ポリフルオレン誘導体等の高分子化合物等が挙げられる。前記ホスト化合物は、１種単独で使用してもよいし、２種以上を併用してもよい。
【００３４】
またさらに本発明においては、前記発光層に正孔輸送材や電子輸送材を必要に応じて含ませても良い。
【００３５】
前記正孔輸送材としては、低分子正孔輸送材、高分子正孔輸送材いずれも用いることができ、陽極から正孔を注入する機能、正孔を輸送する機能、陰極から注入された電子を障壁する機能のいずれかを有しているもので有れば限定されることはなく、例えば以下の材料を挙げることができる。
【００３６】
カルバゾール誘導体、トリアゾール誘導体、オキサゾール誘導体、オキサジアゾール誘導体、イミダゾール誘導体、ポリアリールアルカン誘導体、ピラゾリン誘導体、ピラゾロン誘導体、フェニレンジアミン誘導体、アリールアミン誘導体、アミノ置換カルコン誘導体、スチリルアントラセン誘導体、フルオレノン誘導体、ヒドラゾン誘導体、スチルベン誘導体、シラザン誘導体、芳香族第三アミン化合物、スチリルアミン化合物、芳香族ジメチリデン系化合物、ポルフィリン系化合物、ポリシラン系化合物、ポリ（Ｎ－ビニルカルバゾール）誘導体、アニリン系共重合体、チオフェンオリゴマー、ポリチオフェン等の導電性高分子オリゴマー、ポリチオフェン誘導体、ポリフェニレン誘導体、ポリフェニレンビニレン誘導体、ポリフルオレン誘導体等の高分子化合物等が挙げられる。これらは、１種単独で使用してもよいし、２種以上を併用してもよい。
【００３７】
前記電子輸送材としては電子を輸送する機能、陽極から注入された正孔を障壁する機能のいずれかを有しているもので有れば制限されることはなく例えば以下の材料を挙げることができる。トリアゾール誘導体、オキサゾール誘導体、オキサジアゾール誘導体、フルオ

レノン誘導体、アントラキノジメタン誘導体、アントロン誘導体、ジフェニルキノン誘導体、チオピランジオキシド誘導体、カルボジイミド誘導体、フルオレニリデンメタン誘導体、ジスチリルピラジン誘導体、ナフタレンペリレン等の複素環テトラカルボン酸無水物、フタロシアニン誘導体、８－キノリノール誘導体の金属錯体やメタルフタロシアニン、ベンゾオキサゾールやベンゾチアゾールを配位子とする金属錯体に代表される各種金属錯体、アニリン系共重合体、チオフェンオリゴマー、ポリチオフェン等の導電性高分子オリゴマー、ポリチオフェン誘導体、ポリフェニレン誘導体、ポリフェニレンビニレン誘導体、ポリフルオレン誘導体等の高分子化合物を挙げることができる。
さらに本発明においては発光層に必要に応じて、電気的に不活性なポリマーバインダーを用いることができる。

【００３８】

必要に応じて用いられる電気的に不活性なポリマーバインダーとしては、例えば、ポリ塩化ビニル、ポリカーボネート、ポリスチレン、ポリメチルメタクリレート、ポリブチルメタクリレート、ポリエステル、ポリスルホン、ポリフェニレンオキシド、ポリブタジエン、炭化水素樹脂、ケトン樹脂、フェノキシ樹脂、ポリアミド、エチルセルロース、酢酸ビニル、ＡＢＳ樹脂、ポリウレタン、メラミン樹脂、不飽和ポリエステル、アルキド樹脂、エポキシ樹脂、シリコン樹脂、ポリビニルブチラール、ポリビニルアセタール等を挙げることができる。前記発光層が前記ポリマーバインダーを含有していると、該発光層を湿式製膜法により容易にかつ大面積に塗布形成することができる点で有利である。

【００３９】

前記発光層の厚みとしては、１０～２００ｎｍが好ましく、２０～８０ｎｍがより好ましい。前記厚みが、２００ｎｍを越えると駆動電圧が上昇することがあり、１０ｎｍ未満であると該発光素子が短絡することがある。

【００４０】

－－電子輸送層－－

本発明においては必要に応じて、電子輸送材を含む電子輸送層を設けることができる。前記電子輸送材としては電子を輸送する機能、陽極から注入された正孔を障壁する機能のいずれかを有しているもので有れば制限されることはなく前記電子輸送材を好適に用いることができる。

【００４１】

また、前記電子輸送層には、必要に応じて電気的に不活性なポリマーバインダーを用いることができ、前記ポリマーバインダーを用いることができる。前記電子輸送層の厚みとしては、１０～２００ｎｍが好ましく、２０～８０ｎｍがより好ましい。前記厚みが、２００ｎｍを越えると駆動電圧が上昇することがあり、１０ｎｍ未満であると該発光素子が短絡することがある。

【００４２】

－－正孔輸送層－－

本発明においては必要に応じて、正孔輸送材を含む正孔輸送層を設けることができる。前記正孔輸送材としては正孔を輸送する機能、陰極から注入された電子を障壁する機能のいずれかを有しているもので有れば制限されることはなく前記正孔輸送材を好適に用いることができる。

【００４３】

また、前記正孔輸送層には、必要に応じて電気的に不活性なポリマーバインダーを用いることができ、前記ポリマーバインダーを用いることができる。前記正孔輸送層の厚みとしては、１０～２００ｎｍが好ましく、２０～８０ｎｍがより好ましい。前記厚みが、２００ｎｍを越えると駆動電圧が上昇することがあり、１０ｎｍ未満であると該発光素子が短絡することがある。

【００４４】

－－有機層の形成－－

本発明において、少なくとも一層の有機層は、湿式製膜法で形成される。他の有機層は、

乾式製膜法、湿式製膜法いずれの方法で製膜してもよい。乾式製膜法としては蒸着法やスパッタ法等が挙げられる。湿式製膜法としてはディッピング、スピンコート法、ディップコート法、キャスト法、ダイコート法、ロールコート法、バーコート法、グラビアコート法等のが挙げられ、いずれによっても好適に製膜することができる。

なかでも、前記湿式製膜法による塗布形成の場合、前記有機層を容易に大面積化することができ、高輝度で発光効率に優れた発光素子が低コストで効率よく得られる点で有利である。なお、これらの製膜法の種類の選択は、該有機層の材料に応じて適宜行うことができる。また、湿式製膜法で２層以上積層する場合には、多層との混合を避けるために転写法や印刷法を好適に用いることもできる。

【００４５】

前記湿式製膜法により製膜した場合は、製膜した後、適宜乾燥を行うことができ、該乾燥の条件としては特に制限はないが、塗布形成した層が損傷しない範囲の温度等を採用することができる。

【００４６】

なお、前記塗布液における固形分量溶剤に対する固形分量としては、特に制限はなく、その粘度も湿式製膜方法に応じて任意に選択することができる。

【００４７】

－基材－

本発明において、基材として用いられる材料としては、具体的には例えば、ＹＳＺ（ジルコニア安定化イットリウム）、ガラス等の無機材料、ポリエチレンテレフタレート、ポリブチレンテレフタレート、ポリエチレンナフタレート等のポリエステル、ポリスチレン、ポリカーボネート、ポリエーテルスルホン、ポリアリレート、アリルジグリコールカーボネート、ポリイミド、ポリシクロオレフィン、ノルボルネン樹脂、ポリ（クロロトリフルオロエチレン）、テフロン、ポリテトラフルオロエチレンーポリエチレン共重合体等の高分子材料、などが挙げられる。なかでも、フレキシブルな発光素子や、塗布型発光素子の場合には、高分子材料であることが好ましい。

【００４８】

高分子材料のなかでも、　酸素透過性、透明性、耐熱性、寸法安定性、耐溶剤性、電気絶縁性、加工性、低通気性、低吸湿性等の観点から、ポリエステル、又はポリカーボネート、又はポリエーテルスルホン、又はポリ（クロロトリフルオロエチレン）、テフロン、ポリテトラフルオロエチレンーポリエチレン共重合体等のフッ素原子を含む高分子材料であることが好ましい。

【００４９】

前記基材の形状、構造、大きさ等については、特に制限はなく、発光素子の用途、目的等に応じて適宜選択することができる。一般的には、前記形状としては、板状である。前記構造としては、単層構造であってもよいし、積層構造であってもよく、また、単一部材で形成されていてもよいし、２以上の部材で形成されていてもよい。

【００５０】

前記基材は、無色透明であってもよいし、有色透明であってもよいが、前記発光層から発せられる光を散乱あるいは減衰等させることがない点で、無色透明であるのが好ましい。

【００５１】

前記基材には、その表面又は裏面（前記透明電極側）に透湿防止層（ガスバリア層）を設けることができる。前記透湿防止層（ガスバリア層）の材料としては、窒化珪素、酸化珪素などの無機物が好適に用いられる。該透湿防止層（ガスバリア層）は、例えば、高周波スパッタリング法などにより形成することができる。前記基材には、さらに必要に応じて、ハードコート層、アンダーコート層などを設けてもよい。

【００５２】

－透明電極－

前記透明電極としては、通常、前記有機層に正孔を供給する陽極としての機能を有していればよく、その形状、構造、大きさ等については特に制限はなく、発光素子の用途、目的

に応じて、公知の電極の中から適宜選択することができる。前記透明電極を陰極として機能させることもでき、この場合、前記背面電極を陽極として機能させるようにすればよい。

【００５３】

前記透明電極の材料としては、例えば、金属、合金、金属酸化物、有機導電性化合物、又はこれらの混合物を好適に挙げられ、仕事関数が４．０ｅＶ以上の材料が好ましい。具体例としては、アンチモンやフッ素等をドープした酸化錫（ＡＴＯ、ＦＴＯ）、酸化錫、酸化亜鉛、酸化インジウム、酸化インジウム錫（ＩＴＯ）、酸化亜鉛インジウム（ＩＺＯ）等の半導性金属酸化物、金、銀、クロム、ニッケル等の金属、さらにこれらの金属と導電性金属酸化物との混合物又は積層物、ヨウ化銅、硫化銅などの無機導電性物質、ポリアニリン、ポリチオフェン、ポリピロールなどの有機導電性材料、及びこれらとＩＴＯとの積層物などが挙げられる。

【００５４】

前記透明電極は例えば、印刷方式、コーティング方式等の湿式方式、真空蒸着法、スパッタリング法、イオンプレーティング法等の物理的方式、ＣＶＤ、プラズマＣＶＤ法等の化学的方式、などの中から前記材料との適性を考慮して適宜選択した方法に従って前記基板上に形成することができる。例えば、前記透明電極の材料として、ＩＴＯを選択する場合には、該透明電極の形成は、直流あるいは高周波スパッタ法、真空蒸着法、イオンプレーティング法等に従って行うことができる。また前記透明電極の材料として有機導電性化合物を選択する場合には湿式製膜法に従って行うことができる。

【００５５】

前記透明電極の前記発光素子における形成位置としては、特に制限はなく、該発光素子の用途、目的に応じて適宜選択することができるが、前記基板上に形成されるのが好ましい。この場合、該透明電極は、前記基板における一方の表面の全部に形成されていてもよく、その一部に形成されていてもよい。

【００５６】

なお、前記透明電極のパターニングは、フォトリソグラフィーなどによる化学的エッチングにより行ってもよいし、レーザーなどによる物理的エッチングにより行ってもよく、また、マスクを重ねて真空蒸着やスパッタ等をして行ってもよいし、リフトオフ法や印刷法により行ってもよい。

【００５７】

前記透明電極の厚みとしては、前記材料により適宜選択することができ、一概に規定することはできないが、通常１０ｎｍ～５０μｍであり、５０ｎｍ～２０μｍが好ましい。前記透明電極の抵抗値としては、10^3Ω／□以下が好ましく、10^2Ω／□以下がより好ましい。前記透明電極は、無色透明であっても、有色透明であってもよく、該透明電極側から発光を取り出すためには、その透過率としては、６０％以上が好ましく、７０％以上がより好ましい。この透過率は、分光光度計を用いた公知の方法に従って測定することができる。

【００５８】

なお、前記透明電極については、沢田豊監修「透明電極膜の新展開」シーエムシー刊（１９９９）に詳述があり、これらを本発明に適用することができる。耐熱性の低いプラスティック基材を用いる場合は、ＩＴＯ又はＩＺＯを使用し、１５０℃以下の低温で製膜した透明電極が好ましい。

【００５９】

－－背面電極－－

前記背面電極としては、通常、前記有機層に電子を注入する陰極としての機能を有していればよく、その形状、構造、大きさ等については特に制限はなく、発光素子の用途、目的に応じて、公知の電極の中から適宜選択できる。

前記背面電極を陽極として機能させることもでき、この場合、前記透明電極を陰極として機能させるようにすればよい。

【００６０】

前記背面電極の材料としては、例えば、金属、合金、金属酸化物、電気伝導性化合物、これらの混合物などが挙げられ、仕事関数が４．５ｅＶ以下のものが好ましい。具体例としてはアルカリ金属（たとえば、Ｌｉ、Ｎａ、Ｋ、Ｃｓ等）、アルカリ土類金属（たとえばＭｇ、Ｃａ等）、金、銀、鉛、アルミニウム、ナトリウムーカリウム合金、リチウムーアルミニウム合金、マグネシウムー銀合金、インジウム、イッテルビウム等の希土類金属、などが挙げられる。これらは、１種単独で使用してもよいが、安定性と電子注入性とを両立させる観点からは、２種以上を好適に併用することができる。

【００６１】

これらの中でも、電子注入性の点で、アルカリ金属やアルカリ度類金属が好ましく、保存安定性に優れる点で、アルミニウムを主体とする材料が好ましい。

前記アルミニウムを主体とする材料とは、アルミニウム単独、又はアルミニウムと０．０１～１０重量%のアルカリ金属若しくはアルカリ土類金属との合金若しくは混合物（例えば、リチウムーアルミニウム合金、マグネシウムーアルミニウム合金など）をいう。

【００６２】

なお、前記背面電極の材料については、特開平２－１５５９５号公報、特開平５－１２１１７２号公報に詳述されている。

【００６３】

前記背面電極の形成法は、特に制限はなく、公知の方法に従って行うことができる。例えば、印刷方式、コーティング方式等の湿式方式、真空蒸着法、スパッタリング法、イオンプレーティング法等の物理的方式、ＣＶＤ、プラズマＣＶＤ法等の化学的方式、などの中から前記材料との適性を考慮して適宜選択した方法に従って前記基板上に形成することができる。例えば、前記背面電極の材料として、金属等を選択する場合には、その１種又は２種以上を同時又は順次にスパッタ法等に従って行うことができる。

【００６４】

なお、前記背面電極のパターニングは、フォトリソグラフィーなどによる化学的エッチングにより行ってもよいし、レーザーなどによる物理的エッチングにより行ってもよく、また、マスクを重ねて真空蒸着やスパッタ等をして行ってもよいし、リフトオフ法や印刷法により行ってもよい。

【００６５】

前記背面電極の前記発光素子における形成位置としては、特に制限はなく、該発光素子の用途、目的に応じて適宜選択することができるが、前記有機層上に形成されるのが好ましい。この場合、該背面電極は、前記有機層上の全部に形成されていてもよく、その一部に形成されていてもよい。また、前記背面電極と前記有機層との間に前記アルカリ金属又は前記アルカリ土類金属のフッ化物等による誘電体層を０．１～５ｎｍの厚みで挿入してもよい。なお、該誘電体層は、例えば、真空蒸着法、スパッタリング法、イオンプレーティング法等により形成することができる。

【００６６】

前記透明電極の厚みとしては、前記材料により適宜選択することができ、一概に規定することはできないが、通常１０ｎｍ～５μｍであり、５０ｎｍ～１μｍが好ましい。前記背面電極は、透明であってもよいし、不透明であってもよい。なお、透明な背面電極は、前記背面電極の材料を１～１０ｎｍの厚みに薄く製膜し、更に前記ＩＴＯやＩＺＯ等の透明な導電性材料を積層することにより形成することができる。

【００６７】

－その他の層－

前記その他の層としては、特に制限はなく、目的に応じて適宜選択することができ、例えば、保護層などが挙げられる。前記保護層としては、例えば、特開平７－８５９７４号公報、同７－１９２８６６号公報、同８－２２８９１号公報、同１０－２７５６８２号公報、同１０－１０６７４６号公報等に記載のものが好適に挙げられる。

【００６８】

前記保護層は、前記積層体素子において、その最表面に、例えば、前記基材、前記透明電極、前記有機層、及び前記背面電極がこの順に積層される場合には、該背面電極上に形成され、前記基材、前記背面電極、前記有機層、及び前記透明電極がこの順に積層される場合には、該透明電極上に形成される。

【００６９】

前記保護層の形状、大きさ、厚み等については、適宜選択することができ、その材料としては、水分や酸素等の発光素子を劣化させ得るものを該発光素子内に侵入乃至透過させるのを抑制する機能を有すれば特に制限はなく、例えば、酸化珪素、二酸化珪素、酸化ゲルマニウム、二酸化ゲルマニウム等が挙げられる。

【００７０】

前記保護層の形成方法としては、特に限定はなく、例えば、真空蒸着法、スパッタリング法、反応性スパッタリング法、分子センエピタキシ法、クラスターイオンビーム法、イオンプレーティング法、プラズマ重合法、プラズマＣＶＤ法、レーザーＣＶＤ法、熱ＣＶＤ法、コーティング法、などが挙げられる。

【００７１】

更に、本発明においては、前記発光素子における各層への水分や酸素の侵入を防止する目的で、封止層を設けるのも好ましい。前記封止層の材料としては、例えば、テトラフルオロエチレンと少なくとも１種のコモノマーとを含む共重合体、共重合主鎖に環状構造を有する含フッ素共重合体、ポリエチレン、ポリプロピレン、ポリメチルメタクリレート、ポリイミド、ポリユリア、ポリテトラフルオロエチレン、ポリクロロトリフルオロエチレン、ポリジクロロジフルオロエチレン、クロロトリフルオロエチレン及びジクロロジフルオロエチレンから選択される２種以上の共重合体、吸水率１％以上の吸水性物質、吸水率０．１％以下の防湿性物質、Ｉｎ、Ｓｎ、Ｐｂ、Ａｕ、Ｃｕ、Ａｇ、Ａｌ、Ｔｌ、Ｎｉ等の金属、ＭｇＯ、ＳｉＯ、SiO_2、Al_2O_3、ＧｅＯ、ＮｉＯ、ＣａＯ、ＢａＯ、Fe_2O_3、Y_2O_3、TiO_2等の金属酸化物、MgF_2、ＬｉＦ、AlF_3、CaF_2等の金属フッ化物、パーフルオロアルカン、パーフルオロアミン、パーフルオロエーテル等の液状フッ素化炭素、液状フッ素化炭素に水分や酸素を吸着する吸着剤を分散させたもの、などが挙げられる。

【００７２】

また、本発明においては、前記発光素子を外部との水分や酸素の遮断の目的で封止板、封止容器により、封止剤を用いて封止することが好ましい。封止板、封止容器に用いられる材質としては、ガラス、ステンレス、アルミ等の金属、ポリ（クロロトリフルオロエチレン）、ポリエステル、ポリカーボネート等のプラスティックやセラミック等を用いることができる。封止材としては紫外線硬化樹脂、熱硬化樹脂、二液型硬化樹脂いずれも用いることができる。

【００７３】

本発明においては、封止容器と発光素子の間の空間に水分吸収剤又は不活性液体を設置できる。水分吸収剤としては、特に限定されることはないが例えば酸化バリウム、酸化ナトリウム、酸化カリウム、酸化カルシウム、硫酸ナトリウム、硫酸カルシウム、硫酸マグネシウム、五酸化燐、塩化カルシウム、塩化マグネシウム、塩化銅、フッ化セシウム、フッ化ニオブ、臭化カルシウム、臭化バナジウム、モレキュラーシーブ、ゼオライト、酸化マグネシウム等を挙げることができる。不活性液体としては、特に限定されることはないが例えば、パラフィン類、流動パラフィン類、パーフルオロアルカンやパーフルオロアミン、パーフルオロエーテル等のフッ素系溶剤、塩素系溶剤、シリコーンオイル類が挙げられる。

【００７４】

本発明の発光素子は、前記透明電極と前記背面電極との間に直流（必要に応じて交流成分を含んでもよい）電圧（通常２ボルト～４０ボルト）、又は直流電流を印加することにより、発光を得ることができる。

【００７５】

本発明の発光素子の駆動については、特開平２－１４８６８７号、同６－３０１３５５号、同５－２９０８０号、同７－１３４５５８号、同８－２３４６８５号、同２４１０４７号、米国特許５８２８４２９号、同６０２３３０８号、日本特許第２７８４６１５号等に記載の方法を利用することができる。

【００７６】

【実施例】

以下に、本発明の発光素子の実施例について説明するが、本発明はこれら実施例により何ら限定されない。記載がない限り、実施例の塗布液の調製及び有機層の形成は、水分５０ｐｐｍ以下の窒素置換グローブボックス内で行った。

【００７７】

（実施例１－１）

基材として厚みが０．２ｍｍガラス板を２．５ｃｍ角に切断し、真空チャンバー内に導入し、SnO_2含有率が１０重量％であるＩＴＯターゲットを用いて、ＤＣマグネトロンスパッタ（条件：基材温度１００℃、酸素圧1×10^{-3}Ｐａ）により、透明電極としてのＩＴＯ薄膜（厚み０．２μｍ）を形成した。ＩＴＯ薄膜の表面抵抗は１０Ω／□であった。

【００７８】

次に、前記透明電極を形成した基板を洗浄容器に入れ、ＩＰＡ洗浄した後、これにＵＶーオゾン処理を３０分おこなった。そして、該透明電極の表面に、ポリ（エチレンジオキシチオフェン）・ポリスチレンスルホン酸水分散物（ＢＡＹＥＲ社製、Ｂａｙｔｒｏｎ　Ｐ：固形分１．３％）をスピンコートした後、１５０℃、２時間真空乾燥して厚みが１００ｎｍの正孔注入層を形成した。

【００７９】

一方、直径３ｃｍ、長さ５０ｃｍのガラス製カラム管に活性アルミナ（２００メッシュ、和光純薬工業製）を１００ｇ入れ、このカラムを用いて発光層塗布液の溶剤である１，２－ジクロロエタン（高速液体クロマトグラフ用、和光純薬工業製）５０ｍｌを流速１０ｍｌ／分で流して、精製をおこなった。

【００８０】

１，２－ジクロロエタンの上記精製処理をおこなってから２時間後に、正孔輸送材兼ホスト材としてのポリビニルカルバゾール（Ｍｗ＝６３０００、アルドリッチ製）と燐光発光材であるオルトメタル化錯体としてのトリス（２－フェニルピリジン）イリジウム錯体と電子輸送材である２－（４－ビフェニリル）－５－（４－ｔ－ブチルフェニル）－１，３，４－オキサジアゾール（ＰＢＤ）を４０：１：１２の重量比で上記精製ジクロロエタンに溶解して発光層塗布液を調製した。

【００８１】

該塗布液をスピンコーターを用いて前記正孔注入層の上に塗布し、室温で乾燥させることにより厚みが１００ｎｍの発光層を形成した。

さらにこの発光層上にパターニングしたマスク（発光面積が５ｍｍ×５ｍｍとなるマスク）を設置し、蒸着装置内でマグネシウム：銀＝１０：１（モル比）を０．２５μｍ蒸着し、銀を０．３μｍ蒸着して背面電極を形成した。

前記透明電極（陽極として機能する）及び前記背面電極より、それぞれアルミニウムのリード線を結線し、積層構造体を形成した。

【００８２】

ここで得られた積層構造体を、窒素ガスで置換したグローブボックス内に入れ、ガラス製の封止容器で紫外線硬化型接着剤（長瀬チバ製、ＸＮＲ５４９３）を用いて封止した。以上により、実施例１の発光素子を作成した。

【００８３】

（実施例１－２）

実施例１－１において、発光層の溶剤を１、２－ジクロロエタンの替わりにクロロホルム／メチルエチルケトン（７０／３０体積％）を用いる以外は実施例１－１と同じ方法で精

製し、発光素子を作製、評価した。

【００８４】

（実施例１－３）

実施例１－１において、発光層の溶剤である１，２－ジクロロエタンをカラム精製し２時間後に塗布液調整する替わりに、３日後に塗布液調製する以外は、実施例１－１と同じ方法で、発光素子を作製、評価した。

【００８５】

（実施例１－４）

実施例１－１において、発光層の溶剤である１，２－ジクロロエタンをカラム精製し２時間後に塗布液調整する替わりに、７日後に塗布液調製する以外は、実施例１－１と同じ方法で、発光素子を作製、評価した。

【００８６】

（比較例１－１）

実施例１－１において、発光層の溶剤である１，２－ジクロロエタンをカラム精製しない以外は、実施例１－１と同じ方法で、発光素子を作製、評価した。

【００８７】

（発光素子の評価方法）

上記実施例及び比較例での発光素子を、東洋テクニカ製ソースメジャーユニット２４００型を用いて、直流電圧を有機ＥＬ素子に印加し発光させた。その時の最高輝度をＬｍａｘ、Ｌｍａｘが得られた時の電圧をV_{max}とした。また２００Ｃｄ／ｍ^{2}時の発光効率（η_{200}）を表１に示した。さらに該素子を初期輝度２００Ｃｄ／ｍ^{2}で定電流連続駆動し、輝度が半分になった時間（半減期）を$T_{1/2}$とし、表１に示した。

【００８８】

【表１】

	L_{max} (cd／m^2)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例1-1	88000	15	13.2	12000
実施例1-2	78000	15	12.3	11000
実施例1-3	69000	15	12.1	10000
実施例1-4	65000	15	11.4	9500
比較例1-1	42000	17	8.1	4800

【００８９】

表より、比較例１－１での塗布液では溶剤を精製しておらず、実施例１，２、３，４に比べて、発光輝度、発光効率、耐久性が劣っていることがわかる。

【００９０】

（実施例２－１）

実施例１－１と同様にして、透明電極（ＩＴＯ薄膜）、及び正孔注入層を有する基材を作成した。

【００９１】

５００ｍｌのなす型フラスコに発光層塗布液の溶剤である１，２－ジクロロエタン（高速液体クロマトグラフ用、和光純薬工業製）３００ｍｌ、及び無水硫酸ナトリウム１００ｇを入れ、一昼夜放置し乾燥した。乾燥後濾過し、さらに蒸留をおこなって脱水処理（水分

量３０ｐｐｍ）を行った。脱水処理をした１，２－ジクロロエタンは水分量５０ｐｐｍの窒素ガスグローブボックス内で保存した。

【００９２】

１，２－ジクロロエタンの上記脱水処理をおこなってから２時間後に、正孔輸送材兼ホスト材としてのポリビニルカルバゾール（Ｍｗ＝６３０００、アルドリッチ製）と燐光発光材であるオルトメタル化錯体としてのトリス（２－フェニルピリジン）イリジウム錯体と電子輸送材である２－（４－ビフェニリル）－５－（４－ｔ－ブチルフェニル）－１，３，４－オキサジアゾール（ＰＢＤ）を４０：１：１２の重量比で上記脱水１，２－ジクロロエタンに、水分量５０ｐｐｍの窒素ガスグローブボックス内で溶解して発光層塗布液を調製した。

【００９３】

該塗布液を水分量５０ｐｐｍの窒素ガスグローブボックス内でスピンコーターを用いて前記正孔注入層の上に塗布し、室温で乾燥させることにより厚みが１００ｎｍの発光層を形成した。

【００９４】

実施例１－１と同様にして、発光層上に背面電極を形成し、前記透明電極（陽極として機能する）及び前記背面電極より、それぞれアルミニウムのリード線を結線し、積層構造体を形成した。

【００９５】

ここで得られた積層構造体を、水分量５０ｐｐｍの窒素ガスグローブボックス内で封止する以外は、実施例１－１と同様にして発光素子を作成した。下記表中の発光素子の評価項目は実施例１－１のそれと同じである。

【００９６】

（実施例２－２）

実施例２－１において、発光層の溶剤を１、２－ジクロロエタンの替わりにクロロホルム／メチルエチルケトン（７０／３０体積％；水分量５０ｐｐｍ）を用いる以外は実施例２－１と同じ方法で精製し、発光素子を作製、評価した。

【００９７】

（実施例２－３）

実施例２－１において、発光層の溶剤である１，２－ジクロロエタンを脱水処理し２時間後に塗布液調整する替わりに、７日後に塗布液調製する以外は、実施例２－１と同じ方法で、発光素子を作製、評価した。７日後の塗布液の水分量は９０ｐｐｍであった。

【００９８】

（比較例２－１）

実施例２－１において、発光層の溶剤である１，２－ジクロロエタンを脱水処理しない以外は、実施例２－１と同じ方法で、発光素子を作製、評価した。なお脱水処理しない１，２－ジクロロエタンの水分量は８００ｐｐｍであった。

【００９９】

上記発光素子の評価は、上記実施例（１－１）と同様に行った。

【０１００】

【表２】

	L_{max} (cd／m²)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例2-1	78000	15	12.2	11000
実施例2-2	72000	15	12.3	12000
実施例2-3	59000	15	11.0	9000
比較例2-1	40000	17	7.9	4200

【０１０１】
上記表より、比較例２－１での塗布液では溶剤を脱水処理しておらず、実施例２－１，２－２、２－３に比べて、発光輝度、発光効率、耐久性が劣っている。
【０１０２】
（実施例３－１）
実施例１－１と同様にして、ガラス基板上にＩＴＯ薄膜を形成し、次に正孔注入層を形成した。一方、実施例１－１と同様にして、１，２－ジクロロエタンをカラム精製し、さらにこの１，２－ジクロロエタンを実施例２－１と同様にして脱水処理し、水分量１０ｐｐｍの１，２－ジクロロエタンを得た。精製された１，２－ジクロロエタンを用いて、実施例２－１と同様にして発光層塗布液を調製、発光層形成、積層、封止して発光素子を作成した。下記表中の評価項目は実施例１と同じである。
【０１０３】
【表３】

	L_{max} (cd／m²)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例3-1	96000	14	15.3	14000

【０１０４】
表より、溶剤の脱水処理及びカラム精製との組合せは、単独の精製処理の場合よりも、高発光輝度、低電圧駆動、高発光効率、耐久性に優れることが分かる。
【０１０５】
【発明の効果】
本発明によると、特殊な装置等を必要とせず、高輝度、低電圧駆動、発光効率が極めて高く、耐久性が優れ、低コストな発光素子の製造方法を提供できる。

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(54) METHOD OF MANUFACTURING LIGHT EMITTING ELEMENT

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a method of manufacturing a light emitting element having a high luminance, an excellent light emitting efficiency and durability.
SOLUTION: The light emitting element includes at least one organic layer containing a light emitting layer. The method of manufacturing light emitting element is characterized in that at least one organic layer is formed by wet film forming, by using a compound contained in the organic layer and a coating liquid containing at least one type of solvent refined by at least one refining process.

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CLAIMS

[Claim(s)]
[Claim 1]
The manufacture approach of the light emitting device which forms much more organic layer by wet film production at least using the coating liquid containing at least the compound which is the light emitting device which has much more organic layer containing a luminous layer at least, and is contained in an organic layer, and a kind of solvent refined by at least one purification processing.
[Claim 2]
The manufacture approach according to claim 1 that at least one purification processing is at least one of dehydration part processing and the column purification.
[Claim 3]
The manufacture approach according to claim 2 that at least one purification processing is dehydration part processing and column purification.
[Claim 4]
The manufacture approach according to claim 1 to 3 that the refined moisture content of at least a kind of solvent is 100 ppm or less.
[Claim 5]
The manufacture approach according to claim 1 to 4 that a kind of refined solvent is a kind of halogen system solvent at least.
[Claim 6]
The manufacture approach according to claim 1 to 5 that coating liquid contains phosphorescence luminescent material.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]
This invention relates to the manufacture approach of a light emitting device (organic electroluminescence devices) available in the field of a back light, a flat-panel display, the source of the illumination light, a display device, electrophotography, organic-semiconductor laser, the record light source, the exposure light source, the reading light source, an indicator, a signboard, an optical-communication device, etc.
[0002]
[Description of the Prior Art]
The light emitting device formed by the wet producing-film method is inferior in luminous efficiency and endurance compared with a vacuum evaporationo mold component in each luminescence gestalt of the light emitting device (phosphorescence light emitting device) using a triplet exciton, and the light emitting device (fluorescence light emitting device) using a singlet exciton. The light emitting device formed by the wet producing-film method is cheap, and has the big merit that a large area is possible. A light emitting device by the wet producing-film method which was excellent in luminous efficiency and endurance is desired.
[0003]
In the field of a light emitting device, by manufacture with a vacuum evaporationo mold component, since sublimation purification was carried out, the organic compound did not need to refine. In JP,2001-214159,A, setting to 0.01 ppm or less ionicity high impurity concentration of the sodium of the thin film which consists degradation by generation of heat of this luminescent organic compound by performing purification processing of multiple times to a luminescent organic compound for the purpose of control ****, or a potassium is indicated.
[0004]
[Problem(s) to be Solved by the Invention]
This invention aims at offering the manufacture approach of a light emitting device of having solved said many problems, having excelled in luminous efficiency and luminescence brightness, and having excelled in endurance.
[0005]
[Means for Solving the Problem]
Not in purification processing of an organic compound but in this invention, this invention person found having a problem in the coating liquid in the manufacture approach of the light emitting device by the wet producing-film method, and found out said The means for solving a technical problem. namely
1. Manufacture approach of light emitting device which forms much more organic layer by wet film production at least using coating liquid containing at least compound which is light emitting device which has much more organic layer containing luminous layer at least, and is contained in organic layer, and a kind of solvent refined by at least one purification processing.
2. Manufacture approach given in said 1 whose at least one purification processing is at least one of dehydration part processing and the column purification.
3. Manufacture approach given in said 2 whose at least one purification processing is dehydration part processing and column purification.
4. Manufacture approach given in either [whose refined moisture content of at least a kind of solvent is 100 ppm or less / said] 1 thru/or 3.
5. Manufacture approach given in either [a kind of refined solvent of whose is a kind of halogen system solvent at least / said] 1 thru/or 4.
6. Manufacture approach given in either [in which coating liquid contains phosphorescence luminescent material / said] 1 thru/or 5.
[0006]
[Embodiment of the Invention]
This invention is a light emitting device which has much more organic layer containing a luminous layer at least, and is the manufacture approach of the light emitting device characterized by forming much more organic layer by wet film production at least using the coating liquid containing at least the compound contained in an organic layer, and a kind of solvent by which purification processing was carried out.
[0007]
In this invention, especially purification processing is not limited, for example, can adopt dehydration part processing, a column method, distillation, the cooling recrystallizing method, etc. It is desirable that at least one purification processing uses at least one of dehydration part processing and the column purification, and it is more desirable that at least one purification processing is dehydration part processing and column purification (concomitant use with dehydration part processing and column purification).
[0008]
Before preparing coating liquid in this invention, it is desirable to perform purification processing of a solvent. A front is less than one week before preparation, and will be less than one day still more preferably the 3rd [less than] day preferably. If it refines before this, the effectiveness of purification will decrease. The luminous efficiency of a light emitting device, endurance, etc. are substantially improvable with purification processing of a solvent.
[0009]

As for the solvent of coating liquid, it is desirable to use a kind of halogen system solvent at least from a soluble point. Especially, a chlorine-based solvent is desirable, for example, can mention a carbon tetrachloride, chloroform, dichloromethane, 1,2-dichloroethane, 1,1-dichloroethane, a chlorobenzene, a dichlorobenzene, etc.

[0010]

In addition to the above-mentioned solvent, it can mix with other solvents enumerated below, and can also use. As other solvents, amide series solvents, such as ethers solvents, such as ester solvents, such as aromatic hydrocarbons solvents, such as alcohols solvents, such as ketones, such as an acetone, a methyl ethyl ketone, and a cyclohexanone, a methanol, ethanol, 1-propanol, 2-propanol, and a butanol, benzene, toluene, and a xylene, methyl acetate, and ethyl acetate, diethylether, a tetrahydrofuran, and dioxane, dimethylacetamide, and dimethylformamide, dimethyl sulfoxide, etc. are mentioned.

[0011]

Although coating liquid is explained in full detail behind, in order to optimize the luminescence engine performance and spreading, it may also contain other components suitably including at least the component used for a kind of organic layer at least, and a kind of solvents (organic compound etc.) for dissolving or distributing it.

[0012]

Especially the approach of dehydration part processing of a solvent is not limited, for example, a drying agent is put in into a solvent, and it leaves it, and can perform dehydration processing by distillation after filtration.

[0013]

Although especially a drying agent is not limited, it can choose suitably anhydrous sodium sulfate, sulfuric anhydride calcium, magnesium sulfate, strontium sulfate, a barium sulfate, the barium oxide, a calcium oxide, magnesium oxide, a molecular sieve, a zeolite, etc. Although it can be used filtering after desiccation with a drying agent, it is desirable to use it, distilling further. As for after desiccation, it is desirable to save under an inert gas ambient atmosphere with a moisture content of 100 ppm or less.

[0014]

The moisture content of at least a kind of solvent in which purification processing was carried out by dehydration part processing and/or column purification has desirable 100 ppm or less, its 50 ppm or less are more desirable, and its 30 ppm or less are still more desirable. The impurities which degrade moisture and/or the luminescence engine performance of a light emitting device are reducible with dehydration part processing and/or column purification of a solvent.

[0015]

In column purification, especially the bulking agent used for a column is not limited, for example, can use suitably silica gel, an alumina, cationic ion exchange resin, anionic ion exchange resin, etc. Moreover, basicity and any neutral and acid bulking agent can be used.

[0016]

As for the amount of a bulking agent, it is desirable that it is 50g or more 1kg or less to 100ml of solvents to refine. If fewer than this, the effectiveness of purification will become small, if [than this] more, the amount of a bulking agent will become useless and productive efficiency will fall. Moreover, as for the flow rate at the time of purification, it is desirable that it is [1ml] the following by 1000ml/above by /. If there are few flow rates than this, productive efficiency will worsen. Moreover, the effectiveness of purification is not small desirable if [than this] more.

[0017]

By carrying out column purification of the chlorine-based solvent, it is surmised that chlorine active species, such as a radical and a cation, are removed. If especially chlorine active species remains in a solvent, the ingredient contained in the organic layer mentioned later will be chlorinated, and it will become the cause of endurance aggravation and effectiveness aggravation.

[0018]

In the manufacture approach of this invention, especially a desirable mode is formation of the luminous layer using the coating liquid containing at least a kind of solvent by which purification processing was carried out, and at least one phosphorescence luminescent material. Purification processing has desirable column purification and its concomitant use with column purification and dehydration part processing is more desirable. An alt.metal-ized metal complex etc. is preferably mentioned so that a phosphorescence luminescence ingredient may be explained in full detail below. As for a solvent, it is desirable that it is the solvent which contains a kind of halogen system solvent at least.

[0019]

especially desirable voice — as for the solvent which can be set like and by which purification processing was carried out, it is desirable that it is the solvent from which impurities, such as chlorine active species, such as a radical and a cation, were removed below to a certain concentration. As for the concentration of the alt.metal-ized metal complex chlorinated in the luminous layer, it is desirable that it is below a certain concentration.

[0020]

(Light emitting device)

The light emitting device in this invention is stated to a detail below. It is the layer which the organic layer of this invention means the layer which can be used for a light emitting device, and an organic layer mainly consists of an organic compound, and specifically contains the compound which has the operation effectiveness as a luminescent organic layer, an electronic transportability organic layer, a hole transportability organic layer, an electronic injection layer, a hole impregnation layer, etc. In addition, in the following, the vocabulary a "derivative" means the compound itself and its derivative, for example, a coumarin derivative means a coumarin and its derivative.

[0021]

— Configuration of an organic layer —

Although there is especially no limit and it can choose suitably according to the application and the object of a light emitting device as a formation location in said light emitting device of said organic layer, it is desirable to be formed between the first electrode and the second electrode. Either a transparent electrode or a back plate is OK as said first electrode and the second electrode. An organic layer is formed in the front face or the whole surface on said transparent electrode or said back plate. About the configuration of said organic layer, magnitude, and thickness, there is especially no limit and it can be suitably chosen according to the object.

[0022]

As concrete lamination, a transparence anode plate / luminous layer / cathode, a transparence anode plate / luminous

layer / electronic transporting bed / cathode, a transparence anode plate / electron hole transporting bed / luminous layer / electronic transporting bed / cathode, a transparence anode plate / electron hole transporting bed / luminous layer / cathode, a transparence anode plate / luminous layer / electronic transporting bed / electronic injection layer / cathode, a transparence anode plate / hole injection layer / electron hole transporting bed / luminous layer / electronic transporting bed / electronic injection layer / cathode, etc. are mentioned.

[0023]

— Luminous layer —

The luminous layer used for this invention may consist of a kind of luminescence material at least, and may also contain electron hole transport material, electronic transport material, and host material if needed. It is not limited especially as luminescence material used for this invention, and if it is a fluorescence luminescence compound or a phosphorescence luminescence compound, it can use.

[0024]

As a fluorescence luminescence compound, for example, a benzooxazole derivative, a benzimidazole derivative, A benzothiazole derivative, a styryl benzene derivative, a polyphenyl derivative, A diphenyl butadiene derivative, a tetra-phenyl butadiene derivative, the North America Free Trade Agreement RUIMIDO derivative, A coumarin derivative, a perylene derivative, a peri non derivative, an OKISA diazo-RU derivative, An aldazine derivative, a PIRARIJIN derivative, a cyclopentadiene derivative, A bis-styryl anthracene derivative, the Quinacridone derivative, a pyrrolo pyridine derivative, A thiadiazolo pyridine derivative, a styryl amine derivative, an aromatic series dimethylidene compound, High molecular compounds, such as various metal complexes represented by the metal complex and rare earth complex of an eight-quinolinol derivative, the poly thiophene derivative, a polyphenylene derivative, a polyphenylene vinylene derivative, and the poly fluorene derivative, etc. are mentioned. These can mix and use a kind or two sorts or more.

[0025]

Although not limited especially as a phosphorescence luminescence compound, an alt.metal-ized metal complex or a porphyrin metal complex is desirable.

[0026]

Said alt.metal-ized metal complex is the generic name of the compound group indicated by Akio Yamamoto work "organic metal chemistry-foundation and application" 150 page, 232 pages, Shokabo Publishing Co., Ltd. (1982 issuance), the "Photochemistry and Photophisics of Coodination Compounds" 71-77 page written by H.Yersin, 135-146 pages and Springer-Verlag (1987 issuance), etc. [-] Said organic layer containing this alt.metal-ized metal complex is advantageous at the point of excelling in luminous efficiency by high brightness.

[0027]

As a ligand which forms an alt.metal-ized metal complex, there are various things, and it is indicated by the above-mentioned reference, and 2-phenyl pyridine derivative, 7, 8-benzoquinoline derivative, 2-(2-thienyl) pyridine derivative, 2-(1-naphthyl) pyridine derivative, 2-phenyl quinoline derivative, etc. are mentioned as a desirable ligand also in it. These derivatives may have a substituent if needed. The alt.metal-ized metal complex may have other ligands other than said ligand.

[0028]

alt.metal-ized metal complex used by this invention Inorg.Chem. 1991 and No. 30, 1685page., ** 1988 and No. 27, 3464page., ** 1994 and No. 33, 545 page .Inorg.Chim.Acta 1991, No. 181, 245 page .J.Organomet.Chem. 1987, No. 335, and 293page.J.Am.Chem.Soc. 1985, No. 107, and 1431page. etc. — It is compoundable by various well-known technique.

[0029]

Also in said alt.metal-ized complex, the compound which emits light from a triplet exciton can use it suitably from a viewpoint of the improvement in luminous efficiency in this invention. Moreover, in a porphyrin metal complex, a porphyrin platinum complex is desirable.

[0030]

The compound of said phosphorescence luminescence may be used by the one-sort independent, and may use two or more sorts together. Moreover, said fluorescence luminescence compound and a phosphorescence luminescence compound may be used simultaneously. In this invention, it is desirable to use said phosphorescence luminescence compound from the point of luminescence brightness and luminous efficiency.

[0031]

As a content in said luminous layer of the compound of said phosphorescence luminescence, there is especially no limit, and it can be suitably chosen according to the object, for example, is 0.1 - 70 % of the weight, and its 1 - 20 % of the weight is desirable. When the content of the compound of said phosphorescence luminescence is not 0.1 - 70 % of the weight, the content effectiveness is enough in the content effectiveness fully not being demonstrated and it being 1 - 20 % of the weight.

[0032]

Said host compound is a compound which has the function for energy transfer to arise from the excitation state to the compound of said phosphorescence luminescence, consequently to make the compound of this phosphorescence luminescence emit light.

[0033]

If it is the compound which can carry out energy transfer of the exciton energy to luminescence material as said host material, there will be especially no limit. According to the object, it can choose suitably. Specifically A carbazole derivative, A triazole derivative, an oxazole derivative, an OKISA diazo-RU derivative, An imidazole derivative, the poly aryl alkane derivative, a pyrazoline derivative, A pyrazolone derivative, a phenylenediamine derivative, an arylamine derivative, an amino permutation chalcone derivative, a styryl anthracene derivative, and full — me — non — a derivative — A hydrazone derivative, a stilbene derivative, a silazane derivative, an aromatic series tertiary-amine compound, A styryl amine compound, an aromatic series dimethylidene system compound, a porphyrin system compound, An anthra quinodimethan derivative, an anthrone derivative, a diphenyl quinone derivative, A thiopyran dioxide derivative, a carbodiimide derivative, a full ORENIRIDEN methane derivative, Heterocycle tetracarboxylic acid anhydrides, such as a JISUCHIRIRU pyrazine derivative and naphthalene perylene, The metal complex and metal phthalocyanine of a phthalocyanine derivative and an eight-quinolinol derivative, The various metal complex polysilane system compounds represented by the metal complex which makes benzooxazole and benzothiazole a ligand, The Pori

(N-vinylcarbazole) derivative, an aniline system copolymer, thiophene oligomer, High molecular compounds, such as conductive polymer oligomer, such as the poly thiophene, the poly thiophene derivative, a polyphenylene derivative, a polyphenylene vinylene derivative, and the poly fluorene derivative, etc. are mentioned. Said host compound may be used by the one-sort independent, and may use two or more sorts together.

[0034]

Furthermore, in this invention, electron hole transport material and electronic transport material may be included in said luminous layer if needed.

[0035]

as said electron hole transport material -- low-molecular electron hole transport material and macromolecule electron hole transport material -- if all can be used, it has the function to pour in an electron hole from an anode plate, the function to convey an electron hole, or the function that carries out the obstruction of the electron poured in from cathode and it is, it will not be limited, for example, the following ingredients can be mentioned.

[0036]

A carbazole derivative, a triazole derivative, an oxazole derivative, an OKISA diazo-RU derivative, An imidazole derivative, the poly aryl alkane derivative, a pyrazoline derivative, A pyrazolone derivative, a phenylenediamine derivative, an arylamine derivative, an amino permutation chalcone derivative, a styryl anthracene derivative, and full -- me -- non -- a derivative -- A hydrazone derivative, a stilbene derivative, a silazane derivative, an aromatic series tertiary-amine compound, A styryl amine compound, an aromatic series dimethylidene system compound, a porphyrin system compound, A polysilane system compound, the Pori (N-vinylcarbazole) derivative, an aniline system copolymer, High molecular compounds, such as conductive polymer oligomer, such as thiophene oligomer and the poly thiophene, the poly thiophene derivative, a polyphenylene derivative, a polyphenylene vinylene derivative, and the poly fluorene derivative, etc. are mentioned. These may be used by the one-sort independent and may use two or more sorts together.

[0037]

If it has the function to convey an electron as said electronic transport material, or the function which carries out the obstruction of the electron hole poured in from the anode plate and is, it will not be restricted, for example, the following ingredients can be mentioned. A triazole derivative, an oxazole derivative, an OKISA diazo-RU derivative, full -- me -- non -- a derivative, an anthra quinodimethan derivative, and an anthrone derivative -- A diphenyl quinone derivative, a thiopyran dioxide derivative, a carbodiimide derivative, Heterocycle tetracarboxylic acid anhydrides, such as a full ORENIRIDEN methane derivative, a JISUCHIRIRU pyrazine derivative, and naphthalene perylene, The metal complex and metal phthalocyanine of a phthalocyanine derivative and an eight-quinolinol derivative, The various metal complexes represented by the metal complex which makes benzooxazole and benzothiazole a ligand, High molecular compounds, such as conductive polymer oligomer, such as an aniline system copolymer, thiophene oligomer, and the poly thiophene, the poly thiophene derivative, a polyphenylene derivative, a polyphenylene vinylene derivative, and the poly fluorene derivative, can be mentioned.

Furthermore in this invention, an inactive polymer-binder can be electrically used for a luminous layer if needed.

[0038]

As an inactive polymer-binder, a polyvinyl chloride, a polycarbonate, polystyrene, polymethylmethacrylate, poly butyl methacrylate, polyester, polysulfone, polyphenylene oxide, polybutadiene, hydrocarbon resin, ketone resin, phenoxy resin, a polyamide, ethyl cellulose, vinyl acetate, ABS plastics, polyurethane, melamine resin, unsaturated polyester, alkyd resin, an epoxy resin, silicon resin, a polyvinyl butyral, a polyvinyl acetal, etc. can be mentioned to the electric target used if needed, for example. If said luminous layer contains said polymer-binder, it is advantageous at the point which can carry out spreading formation of this luminous layer easily by the wet producing-film method at a large area.

[0039]

As thickness of said luminous layer, 10-200nm is desirable and 20-80nm is more desirable. When said thickness exceeds 200nm, driver voltage may go up, and when it is less than 10nm, this light emitting device may short-circuit.

[0040]

-- Electronic transporting bed --

The electronic transporting bed which contains electronic transport material if needed in this invention can be prepared. It will not be restricted, if it has the function to convey an electron as said electronic transport material, or the function which carries out the obstruction of the electron hole poured in from the anode plate and is, and said electronic transport material can be used suitably.

[0041]

Moreover, an inactive polymer-binder can be electrically used for said electronic transporting bed if needed, and said polymer-binder can be used for it. As thickness of said electronic transporting bed, 10-200nm is desirable and 20-80nm is more desirable. When said thickness exceeds 200nm, driver voltage may go up, and when it is less than 10nm, this light emitting device may short-circuit.

[0042]

-- Electron hole transporting bed --

The electron hole transporting bed which contains electron hole transport material if needed in this invention can be prepared. It will not be restricted, if it has the function to convey an electron hole as said electron hole transport material, or the function which carries out the obstruction of the electron poured in from cathode and is, and said electron hole transport material can be used suitably.

[0043]

Moreover, an inactive polymer-binder can be electrically used for said electron hole transporting bed if needed, and said polymer-binder can be used for it. As thickness of said electron hole transporting bed, 10-200nm is desirable and 20-80nm is more desirable. When said thickness exceeds 200nm, driver voltage may go up, and when it is less than 10nm, this light emitting device may short-circuit.

[0044]

-- Formation of an organic layer --

In this invention, much more organic layer at least is formed by the wet producing-film method. other organic layers -- the dry type producing-film method and the wet producing-film method -- a film may be produced by which approach. Vacuum deposition, a spatter, etc. are mentioned as a dry type producing-film method. As a wet producing-film method, those, such as dipping, a spin coat method, a dip coating method, the cast method, the DAIKO-TO method, the

roll coat method, the bar coat method, and the GURABIAKO-TO method, is mentioned, and a film can be suitably produced by all.
The light emitting device which could large-area-ize said organic layer easily, and was excellent in luminous efficiency especially with high brightness in the spreading formation by said wet producing-film method is advantageous at the point efficiently acquired by low cost. In addition, selection of the class of these producing-film methods can be suitably performed according to the ingredient of this organic layer. Moreover, when carrying out a laminating more than two-layer by the wet producing-film method, in order to avoid mixing with a multilayer, a replica method and print processes can also be used suitably.
[0045]
Although it can dry suitably and there is especially no limit as conditions for this desiccation after producing a film when a film is produced by said wet producing-film method, the temperature of the range which the layer which carried out spreading formation does not damage etc. is employable.
[0046]
In addition, as an amount of solid content to the amount solvent of solid content in said coating liquid, there is especially no limit and the viscosity can also choose it as arbitration according to the wet film production approach.
[0047]
- Base material -
In this invention, as an ingredient used as a base material Specifically For example, inorganic materials, such as YSZ (zirconia stabilization yttrium) and glass, Polyester, such as polyethylene terephthalate, polybutylene terephthalate, and polyethylenenaphthalate, Polystyrene, a polycarbonate, polyether sulphone, polyarylate, Polymeric materials, such as allyl compound diethylene glycol carbonate, polyimide, the poly cycloolefin, norbornene resin, Pori (chlorotrifluoroethylene), Teflon, and a polytetrafluoroethylene-polyethylene copolymer, etc. are mentioned. Especially, in the case of a flexible light emitting device and a spreading mold light emitting device, it is desirable that they are polymeric materials.
[0048]
Even inside of polymeric materials, It is desirable that they are the polymeric materials which contain fluorine atoms, such as polyester, a polycarbonate, polyether sulphone or Pori (chlorotrifluoroethylene), Teflon, and a polytetrafluoroethylene-polyethylene copolymer, from viewpoints, such as oxygen permeability, transparency, thermal resistance, dimensional stability, solvent resistance, electric insulation, workability, low permeability, and low hygroscopicity.
[0049]
About the configuration of said base material, structure, and magnitude, there is especially no limit and it can be suitably chosen according to the application of a light emitting device, the object, etc. Generally, as said configuration, it is tabular. As said structure, you may be monolayer structure, and may be a laminated structure, and it may be formed by the single member, and may be formed by two or more members.
[0050]
It is desirable that said base material may be transparent and colorless, and it is transparent and colorless at the point to which dispersion or attenuation does not carry out light emitted from said luminous layer although you may be colored transparence.
[0051]
A moisture permeation prevention layer (gas barrier layer) can be prepared in the front face or rear face (said transparent electrode side) at said base material. As an ingredient of said moisture permeation prevention layer (gas barrier layer), inorganic substances, such as silicon nitride and oxidation silicon, are used suitably. This moisture permeation prevention layer (gas barrier layer) can be formed for example, by a RF-sputtering method etc. A rebound ace court layer, an under coat layer, etc. may be further prepared in said base material if needed.
[0052]
- Transparent electrode -
That what is necessary is just to usually have the function as an anode plate which supplies an electron hole to said organic layer as said transparent electrode, there is especially no limit about the configuration, structure, and magnitude, and it can choose suitably from well-known electrodes according to the application of a light emitting device, and the object. What is necessary is also being able to operate said transparent electrode as cathode and making it just operate said back plate as an anode plate in this case.
[0053]
As an ingredient of said transparent electrode, for example, a metal, an alloy, a metallic oxide, organic conductivity compounds, or such mixture can be mentioned suitably, and an ingredient 4.0eV or more has a desirable work function. the tin oxide (ATO --) which doped antimony, a fluorine, etc. as an example FTO, tin oxide, a zinc oxide, indium oxide, indium oxide tin (ITO), Semiconductance metallic oxides, such as a zinc oxide indium (IZO), gold, silver, chromium, Organic conductivity ingredients, such as inorganic conductivity matter, such as mixture of these metals and conductive metallic oxide or laminated material, copper iodide, and copper sulfide, the poly aniline, the poly thiophene, and polypyrrole, the laminated material of these and ITO, etc. are mentioned to metals, such as nickel, and a pan.
[0054]
Said transparent electrode can be formed on said substrate according to the approach suitably chosen in consideration of fitness with said ingredient from chemical methods, such as physical methods, such as wet methods, such as a printing method and a coating method, vacuum evaporation technique, the sputtering method, and the ion plating method, CVD, and a plasma-CVD method, etc. For example, as an ingredient of said transparent electrode, when choosing ITO, formation of this transparent electrode can be performed according to a direct current or a high frequency spatter, vacuum evaporation technique, the ion plating method, etc. Moreover, when choosing an organic conductivity compound as an ingredient of said transparent electrode, it can carry out according to the wet producing-film method.
[0055]
Although there is especially no limit and it can choose suitably according to the application of this light emitting device, and the object as a formation location in said light emitting device of said transparent electrode, being formed on said substrate is desirable. in this case, also in said substrate, a front face boils all, it may be formed and this transparent electrode may be formed in that part.

[0056]
In addition, the chemical etching by photolithography etc. may perform patterning of said transparent electrode, physical etching by laser etc. may perform it, and it may carry out vacuum deposition, a spatter, etc. in piles, may perform a mask, and may perform it by the lift-off method or print processes.
[0057]
Although it can choose suitably with said ingredient and cannot generally specify as thickness of said transparent electrode, it is usually 10nm - 50 micrometers, and 50nm - 20 micrometers are desirable. As resistance of said transparent electrode, below 103ohms / ** are desirable, and below 102ohms / ** are more desirable. It may be transparent and colorless, or may be colored transparence, and in order to take out luminescence from this transparent electrode side, as the permeability, said 60% or more of transparent electrode is desirable, and is more desirable. [70% or more of] This permeability can be measured according to the well-known approach which used the spectrophotometer.
[0058]
In addition, about said transparent electrode, the Sawada ****** "new expansion of transparent electrode film" CMC ** (1999) has a detailed description, and these can be applied to this invention. When using a heat-resistant low plastic base material, the transparent electrode which used ITO or IZO and produced the film at low temperature 150 degrees C or less is desirable.
[0059]
-- Back plate --
That what is necessary is just to usually have the function as cathode to inject an electron into said organic layer, as said back plate, there is especially no limit about the configuration, structure, and magnitude, and it can choose suitably from well-known electrodes according to the application of a light emitting device, and the object.
What is necessary is also being able to operate said back plate as an anode plate, and making it just operate said transparent electrode as cathode in this case.
[0060]
As an ingredient of said back plate, for example, a metal, an alloy, a metallic oxide, electrical conductivity compounds, such mixture, etc. are mentioned, and a thing 4.5eV or less has a desirable work function. As an example, rare earth metals, such as alkali metal (for example, Li, Na, K, Cs, etc.), alkaline earth metal (for example, Mg, calcium, etc.), gold, silver, lead, aluminum, a sodium-potassium alloy, a lithium-aluminium alloy, a magnesium-silver alloy, an indium, and an ytterbium, etc. are mentioned. Although these may be used by the one-sort independent, they can use two or more sorts together suitably from a viewpoint which reconciles stability and electron injection nature.
[0061]
Also in these, in respect of electron injection nature, alkali metal and an alkalinity metal are desirable and the ingredient which makes aluminum a subject in that it excels in preservation stability is desirable.
The ingredient which makes said aluminum a subject means an alloy or mixture with aluminum independence or aluminum, 0.01 - 10% of the weight of alkali metal, or an alkaline earth metal (for example, a lithium-aluminum alloy, a magnesium-aluminum alloy, etc.).
[0062]
In addition, the ingredient of said back plate is explained by JP,2-15595,A and JP,5-121172,A in full detail.
[0063]
There is no method of forming said back plate, and it can perform especially a limit according to a well-known approach. For example, according to the approach suitably chosen in consideration of fitness with said ingredient from chemical methods, such as physical methods, such as wet methods, such as a printing method and a coating method, vacuum evaporation technique, the sputtering method, and the ion plating method, CVD, and a plasma-CVD method, etc., it can form on said substrate. For example, as an ingredient of said back plate, when choosing a metal etc., its one sort or two sorts or more can be performed to coincidence or sequential according to a spatter etc.
[0064]
In addition, the chemical etching by photolithography etc. may perform patterning of said back plate, physical etching by laser etc. may perform it, and it may carry out vacuum deposition, a spatter, etc. in piles, may perform a mask, and may perform it by the lift-off method or print processes.
[0065]
Although there is especially no limit and it can choose suitably according to the application of this light emitting device, and the object as a formation location in said light emitting device of said back plate, being formed on said organic layer is desirable. In this case, this back plate may be formed in all on said organic layer, and may be formed in that part. Moreover, the dielectric layer by the fluoride of said alkali metal or said alkaline earth metal etc. may be inserted by the thickness of 0.1-5nm between said back plates and said organic layers. In addition, this dielectric layer can be formed by vacuum evaporation technique, the sputtering method, the ion plating method, etc.
[0066]
Although it can choose suitably with said ingredient and cannot generally specify as thickness of said transparent electrode, it is usually 10nm - 5 micrometers, and 50nm - 1 micrometer is desirable. Said back plate may be transparent and may be opaque. In addition, a transparent back plate can produce the ingredient of said back plate thinly in thickness of 1-10nm, and can form it by carrying out the laminating of the conductive ingredient with said still more transparent ITO, IZO, etc.
[0067]
- Other layers -
As a layer of said others, there is especially no limit, and it can be suitably chosen according to the object, for example, a protective layer etc. is mentioned. As said protective layer, the thing of a publication is suitably mentioned to JP,7-85974,A, a 7-192866 official report, a 8-22891 official report, a 10-275682 official report, a 10-106746 official report, etc., for example.
[0068]
In said layered product component, said protective layer is formed on this back plate, when the laminating of said base material, said transparent electrode, said organic layer, and said back plate is carried out to this order, and when the laminating of said base material, said back plate, said organic layer, and said transparent electrode is carried out to this order, it is formed on this transparent electrode in that outermost surface, for example.

[0069]
About the configuration of said protective layer, magnitude, and thickness, it can choose suitably, as the ingredient, if it has the function which controls making what may degrade light emitting devices, such as moisture and oxygen, invade thru/or penetrate in this light emitting device, there will be especially no limit, for example, oxidation silicon, a silicon dioxide, a germanium dioxide, diacid-ized germanium, etc. are mentioned.
[0070]
As the formation approach of said protective layer, there is especially no definition, for example, a vacuum deposition method, the sputtering method, a reactive sputtering method, the molecule SENEPI taxi method, the ionized cluster beam method, the ion plating method, a plasma polymerization method, a plasma-CVD method, a laser CVD method, a heat CVD method, a coating method, etc. are mentioned.
[0071]
Furthermore, in this invention, it is the object which prevents the trespass of the moisture to each class, or oxygen in said light emitting device, and it is also desirable to prepare a closure layer. The copolymer which contains tetrafluoroethylene and at least one sort of comonomers as an ingredient of said closure layer, for example, The fluorine-containing copolymer which has cyclic structure in a copolymerization principal chain, polyethylene, polypropylene, Polymethylmethacrylate, polyimide, polyurea, polytetrafluoroethylene, Polychlorotrifluoroethylene resin, poly dichlorodifluoroethene, Two or more sorts of copolymers chosen from chlorotrifluoroethylene and dichlorodifluoroethene, The absorptivity matter of 1% or more of water absorption, the dampproof matter of 0.1% or less of water absorption, In, Metals, such as Sn, Pb, Au, Cu, Ag, aluminum, Tl, and nickel, MgO, SiO, SiO2, aluminum 2O3, GeO, NiO, CaO, BaO, The metallic oxide of Fe 2O3, Y2O3, and TiO2 grade, MgF2, LiF, The thing which made liquefied fluorination carbon, such as AlF3, a metal fluoride of CaF2 grade, par fluoroalkane, a perfluoro amine, and par full OROE-Tell, and liquefied fluorination carbon distribute the adsorbent which adsorbs moisture and oxygen is mentioned.
[0072]
Moreover, in this invention, it is desirable to close said light emitting device with a closure plate and a closure container using encapsulant for moisture with the exterior or the object of cutoff of oxygen. As construction material used for a closure plate and a closure container, plastics, ceramics, etc., such as metals, such as glass, stainless steel, and aluminum, Pori (chlorotrifluoroethylene), polyester, and a polycarbonate, can be used. as a sealing agent — ultraviolet-rays hardening resin, heat-curing resin, and 2 liquid mold-curing resin — all can be used.
[0073]
In this invention, a water absorption agent or an inactive liquid can be installed in the space between a closure container and a light emitting device. although not limited especially as a water absorption agent — for example, barium-oxide, sodium oxide, potassium oxide, calcium-oxide, sodium-sulfate, calcium-sulfate, magnesium sulfate, phosphorus pentoxide, calcium chloride, magnesium chloride, copper chloride, cesium fluoride, and niobium fluoride, a calcium bromide, and bromination — vanadium, a molecular sieve, a zeolite, a magnesium oxide, etc. can be mentioned. Especially as an inactive liquid, although not limited, fluorine system solvents, such as paraffin, liquid paraffins, par fluoroalkane, and a perfluoro amine, par full OROE-Tell, a chlorine-based solvent, and silicone oil are mentioned.
[0074]
The light emitting device of this invention can obtain luminescence by impressing a direct-current (alternating current component also being included if needed) electrical potential difference (usually 2 volts - 40 volts) or a direct current between said transparent electrodes and said back plates.
[0075]
The approach of a publication can be used for JP,2-148687,A, 6-301355, 5-29080, 7-134558, 8-234685, said 241047 numbers, a U.S. Pat. No. 5828429 number, said 6023308 numbers, the Japanese patent No. 2784615, etc. about actuation of the light emitting device of this invention.
[0076]
[Example]
Although the example of the light emitting device of this invention is explained below, this invention is not limited at all by these examples. As long as it was unstated, preparation of the coating liquid of an example and formation of an organic layer were performed in the nitrogen-purge glove compartment with a moisture of 50 ppm or less.
[0077]
(Example 1-1)
As a base material, thickness cut 0.2mm glass plate on 2.5cm square, and introduced in the vacuum chamber, and SnO2 content formed the ITO thin film (thickness of 0.2 micrometers) as a transparent electrode using the ITO target which is 10 % of the weight by DC magnetron sputtering (conditions: base material temperature of 100 degrees C, oxygen pressure 1x10-3Pa). The surface electrical resistance of an ITO thin film was 10ohm/**.
[0078]
Next, the substrate in which said transparent electrode was formed was put into the wash fountain, and after carrying out IPA washing, UV-ozonization was performed to this for 30 minutes. And it is SUPINKO to the front face of this transparent electrode about Pori (ethylene dioxythiophene) and a polystyrene sulfonate water distribution object (the product made from BAYER, Baytron P: 1.3% of solid content). - After carrying out TO, the vacuum drying was carried out for 2 hours, and 150 degrees C of hole injection layers whose thickness is 100nm were formed.
[0079]
100g (200 meshes, product made from the Wako Pure Chem industry) of activated aluminas was put into glass column tubing with a diameter [of 3cm], and a die length of 50cm, and it refined by on the other hand passing 1,2-dichloroethane (object for high-speed liquid chromatographs, product made from Wako Pure Chem industry) 50ml which is the solvent of luminous layer coating liquid by part for 10ml/of the rates of flow using this column.
[0080]
2 hours after performing the above-mentioned purification processing of 1,2-dichloroethane As an alt.metal-ized complex which is the polyvinyl carbazole (Mw=63000, Aldrich make) and phosphorescence luminescence material as host [electron hole transport material-cum-] material 1, 3, and 4-OKISA diazo-RU (PBD) is dissolved in the above-mentioned purification dichloroethane by the weight ratio of 40:1:12. 2-(4-biphenylyl)-5-(4-t-buthylphenyl)- which is a ** tris (2-phenyl pyridine) iridium complex and electronic transport material — Luminous layer coating liquid was prepared.
[0081]

This coating liquid was applied on said hole injection layer using the spin coater, and thickness formed the luminous layer which is 100nm by making it dry at a room temperature.
Furthermore, the mask (mask with which luminescence area is set to 5mmx5mm) which carried out patterning was installed on this luminous layer, magnesium:silver =10:1 [0.25-micrometer] (mole ratio) was vapor-deposited within vacuum evaporationo equipment, 0.3 micrometers of silver were vapor-deposited, and the back plate was formed. From said transparent electrode (it functions as an anode plate) and said back plate, the lead wire of aluminum was connected, respectively and the laminating structure was formed.

[0082]
The laminating structure obtained here was put in in the glove compartment permuted with nitrogen gas, and was closed using ultraviolet curing mold adhesives (the product made from the Nagase tiba, XNR5493) with the glass closure container. The light emitting device of an example 1 was created by the above.

[0083]
(Example 1-2)
In the example 1-1, except using chloroform/methyl ethyl ketone (70/30 volume %) instead of 1 and 2-dichloroethane, it refined by the same approach as an example 1-1, and the light emitting device was produced and the solvent of a luminous layer was evaluated.

[0084]
(Example 1-3)
In the example 1-1, instead of carrying out column purification of the 1,2-dichloroethane which is the solvent of a luminous layer, and carrying out coating liquid adjustment 2 hours after, it is the same approach as an example 1-1, and the light emitting device was produced and evaluated except carrying out coating liquid preparation three days after.

[0085]
(Example 1-4)
In the example 1-1, instead of carrying out column purification of the 1,2-dichloroethane which is the solvent of a luminous layer, and carrying out coating liquid adjustment 2 hours after, it is the same approach as an example 1-1, and the light emitting device was produced and evaluated except carrying out coating liquid preparation seven days after.

[0086]
(Example 1-1 of a comparison)
In the example 1-1, except not carrying out column purification of the 1,2-dichloroethane which is the solvent of a luminous layer, it is the same approach as an example 1-1, and the light emitting device was produced and evaluated.

[0087]
(The assessment approach of a light emitting device)
Direct current voltage was impressed to the organic EL device, and the light emitting device in the above-mentioned example and the example of a comparison was made to emit light using the source major unit 2400 mold made from Oriental TEKUNIKA. The electrical potential difference when Lmax and Lmax are obtained in the highest brightness at that time was set to Vmax. Moreover, the luminous efficiency at 200 Cd/m 2:00 (eta 200) was shown in a table 1. Furthermore, constant current continuation actuation of this component was carried out by initial brightness 200 Cd/m2, time amount (half-life) from which brightness became half was set to T1/2, and it was shown in a table 1.

[0088]
[A table 1]

	L_{max} (cd／m²)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例1-1	88000	15	13.2	12000
実施例1-2	78000	15	12.3	11000
実施例1-3	69000	15	12.1	10000
実施例1-4	65000	15	11.4	9500
比較例1-1	42000	17	8.1	4800

[0089]
It turns out that a solvent is not refined in the coating liquid in the example 1-1 of a comparison, but luminescence brightness, luminous efficiency, and endurance are inferior to the table compared with examples 1, 2, 3, and 4.

[0090]
(Example 2-1)
The transparent electrode (ITO thin film) and the base material which has a hole injection layer were created like the example 1-1.

[0091]
1,2-dichloroethane (object for high-speed liquid chromatographs, product made from Wako Pure Chem industry) 300ml which is the solvent of luminous layer coating liquid, and 100g of anhydrous sodium sulfate were put into the 500ml

mold flask to make, and it was left one whole day and night, and dried. It filtered after desiccation, it distilled further and dehydration processing (moisture content of 30 ppm) was performed. The 1,2-dichloroethane which carried out dehydration processing was saved in the nitrogen gas globe box with a moisture content of 50 ppm.
[0092]
2 hours after performing the above-mentioned dehydration processing of 1,2-dichloroethane As an alt.metal-ized complex which is the polyvinyl carbazole (Mw=63000, Aldrich make) and phosphorescence luminescence material as host [electron hole transport material-cum-] material 2-(4-biphenylyl)-5-(4-t-buthylphenyl)- which is a ** tris (2-phenyl pyridine) iridium complex and electronic transport material -- 1, 3, and 4-OKISA diazo-RU (PBD) to the above-mentioned dehydration 1,2-dichloroethane by the weight ratio of 40:1:12 It dissolved in the nitrogen gas globe box with a moisture content of 50 ppm, and luminous layer coating liquid was prepared.
[0093]
Thickness formed the luminous layer which is 100nm by applying this coating liquid on said hole injection layer using a spin coater in a nitrogen gas globe box with a moisture content of 50 ppm, and drying it at a room temperature.
[0094]
Like the example 1-1, the back plate was formed on the luminous layer, from said transparent electrode (it functions as an anode plate) and said back plate, the lead wire of aluminum was connected, respectively and the laminating structure was formed.
[0095]
The light emitting device was created like the example 1-1 except closing the laminating structure obtained here in a nitrogen gas globe box with a moisture content of 50 ppm. The evaluation criteria of the light emitting device in the following table are the same as it of an example 1-1.
[0096]
(Example 2-2)
In the example 2-1, except using chloroform/methyl ethyl ketone (70/30 volume %; moisture content of 50 ppm) instead of 1 and 2-dichloroethane, it refined by the same approach as an example 2-1, and the light emitting device was produced and the solvent of a luminous layer was evaluated.
[0097]
(Example 2-3)
In the example 2-1, instead of carrying out dehydration processing of the 1,2-dichloroethane which is the solvent of a luminous layer, and carrying out coating liquid adjustment 2 hours after, it is the same approach as an example 2-1, and the light emitting device was produced and evaluated except carrying out coating liquid preparation seven days after. The moisture content of the coating liquid of seven days after was 90 ppm.
[0098]
(Example 2-1 of a comparison)
In the example 2-1, except not carrying out dehydration processing of the 1,2-dichloroethane which is the solvent of a luminous layer, it is the same approach as an example 2-1, and the light emitting device was produced and evaluated. In addition, the moisture content of the 1,2-dichloroethane which does not carry out dehydration processing was 800 ppm.
[0099]
Assessment of the above-mentioned light emitting device was performed like the above-mentioned example (1-1).
[0100]
[A table 2]

	L_{max} (cd/m^2)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例2-1	78000	15	12.2	11000
実施例2-2	72000	15	12.3	12000
実施例2-3	59000	15	11.0	9000
比較例2-1	40000	17	7.9	4200

[0101]
With the coating liquid in the example 2-1 of a comparison, dehydration processing of the solvent is not carried out, but luminescence brightness, luminous efficiency, and endurance are inferior to the above-mentioned table compared with an example 2-1, 2-2, and 2-3.
[0102]
(Example 3-1)
Like the example 1-1, the ITO thin film was formed on the glass substrate, and then the hole injection layer was formed. On the other hand, like the example 1-1, column purification of the 1,2-dichloroethane was carried out, dehydration processing of this 1,2-dichloroethane was further carried out like the example 2-1, and 1,2-dichloroethane with a moisture content of 10 ppm was obtained. the refined 1,2-dichloroethane -- using -- an example 2-1 -- the same -- carrying out -- luminous layer coating liquid -- preparation, luminous layer formation, and a laminating -- it closed and the light emitting device was created. The evaluation criteria in the following table are the same as an

example 1.
[0103]
[A table 3]

	L_{max} (cd／m²)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例3-1	96000	14	15.3	14000

[0104]
It turns out that the case of purification processing that the combination with dehydration processing of a solvent and column purification is more independent than a table is excelled in high luminescence brightness, low-battery actuation, high luminous efficiency, and endurance.
[0105]
[Effect of the Invention]
According to this invention, special equipment etc. is not needed, but high brightness, low-battery actuation, and luminous efficiency are very high, endurance is excellent, and the manufacture approach of a low cost light emitting device can be offered.

[Translation done.]

TECHNICAL FIELD

[Field of the Invention]
This invention relates to the manufacture approach of a light emitting device (organic electroluminescence devices) available in the field of a back light, a flat-panel display, the source of the illumination light, a display device, electrophotography, organic-semiconductor laser, the record light source, the exposure light source, the reading light source, an indicator, a signboard, an optical-communication device, etc.
[0002]

[Translation done.]

PRIOR ART

[Description of the Prior Art]
The light emitting device formed by the wet producing-film method is inferior in luminous efficiency and endurance compared with a vacuum evaporationo mold component in each luminescence gestalt of the light emitting device (phosphorescence light emitting device) using a triplet exciton, and the light emitting device (fluorescence light emitting device) using a singlet exciton. The light emitting device formed by the wet producing-film method is cheap, and has the big merit that a large area is possible. A light emitting device by the wet producing-film method which was excellent in luminous efficiency and endurance is desired.
[0003]
In the field of a light emitting device, by manufacture with a vacuum evaporationo mold component, since sublimation purification was carried out, the organic compound did not need to refine. In JP,2001-214159,A, setting to 0.01 ppm or less ionicity high impurity concentration of the sodium of the thin film which consists degradation by generation of heat of this luminescent organic compound by performing purification processing of multiple times to a luminescent organic compound for the purpose of control ****, or a potassium is indicated.
[0004]

[Translation done.]

EFFECT OF THE INVENTION

[Effect of the Invention]
According to this invention, special equipment etc. is not needed, but high brightness, low-battery actuation, and luminous efficiency are very high, endurance is excellent, and the manufacture approach of a low cost light emitting device can be offered.

[Translation done.]

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention]
This invention aims at offering the manufacture approach of a light emitting device of having solved said many problems, having excelled in luminous efficiency and luminescence brightness, and having excelled in endurance.
[0005]

[Translation done.]

MEANS

[Means for Solving the Problem]
Not in purification processing of an organic compound but in this invention, this invention person found having a problem in the coating liquid in the manufacture approach of the light emitting device by the wet producing-film method, and found out said The means for solving a technical problem. namely
1. Manufacture approach of light emitting device which forms much more organic layer by wet film production at least using coating liquid containing at least compound which is light emitting device which has much more organic layer containing luminous layer at least, and is contained in organic layer, and a kind of solvent refined by at least one purification processing.
2. Manufacture approach given in said 1 whose at least one purification processing is at least one of dehydration part processing and the column purification.
3. Manufacture approach given in said 2 whose at least one purification processing is dehydration part processing and column purification.
4. Manufacture approach given in either [whose refined moisture content of at least a kind of solvent is 100 ppm or less / said] 1 thru/or 3.
5. Manufacture approach given in either [a kind of refined solvent of whose is a kind of halogen system solvent at least / said] 1 thru/or 4.
6. Manufacture approach given in either [in which coating liquid contains phosphorescence luminescent material / said] 1 thru/or 5.
[0006]
[Embodiment of the Invention]
This invention is a light emitting device which has much more organic layer containing a luminous layer at least, and is the manufacture approach of the light emitting device characterized by forming much more organic layer by wet film production at least using the coating liquid containing at least the compound contained in an organic layer, and a kind of solvent by which purification processing was carried out.
[0007]
In this invention, especially purification processing is not limited, for example, can adopt dehydration part processing, a column method, distillation, the cooling recrystallizing method, etc. It is desirable that at least one purification processing uses at least one of dehydration part processing and the column purification, and it is more desirable that at least one purification processing is dehydration part processing and column purification (concomitant use with dehydration part processing and column purification).
[0008]
Before preparing coating liquid in this invention, it is desirable to perform purification processing of a solvent. A front is less than one week before preparation, and will be less than one day still more preferably the 3rd [less than] day preferably. If it refines before this, the effectiveness of purification will decrease. The luminous efficiency of a light emitting device, endurance, etc. are substantially improvable with purification processing of a solvent.
[0009]
As for the solvent of coating liquid, it is desirable to use a kind of halogen system solvent at least from a soluble point. Especially, a chlorine-based solvent is desirable, for example, can mention a carbon tetrachloride, chloroform, dichloromethane, 1,2-dichloroethane, 1,1-dichloroethane, a chlorobenzene, a dichlorobenzene, etc.
[0010]
In addition to the above-mentioned solvent, it can mix with other solvents enumerated below, and can also use. As other solvents, amide series solvents, such as ethers solvents, such as ester solvents, such as aromatic hydrocarbons solvents, such as alcohols solvents, such as ketones, such as an acetone, a methyl ethyl ketone, and a cyclohexanone, a methanol, ethanol, 1-propanol, 2-propanol, and a butanol, benzene, toluene, and a xylene, methyl acetate, and ethyl acetate, diethylether, a tetrahydrofuran, and dioxane, dimethylacetamide, and dimethylformamide, dimethyl sulfoxide, etc. are mentioned.
[0011]
Although coating liquid is explained in full detail behind, in order to optimize the luminescence engine performance and spreading, it may also contain other components suitably including at least the component used for a kind of organic layer at least, and a kind of solvents (organic compound etc.) for dissolving or distributing it.
[0012]
Especially the approach of dehydration part processing of a solvent is not limited, for example, a drying agent is put in into a solvent, and it leaves it, and can perform dehydration processing by distillation after filtration.
[0013]
Although especially a drying agent is not limited, it can choose suitably anhydrous sodium sulfate, sulfuric anhydride calcium, magnesium sulfate, strontium sulfate, a barium sulfate, the barium oxide, a calcium oxide, magnesium oxide, a molecular sieve, a zeolite, etc. Although it can be used filtering after desiccation with a drying agent, it is desirable to use it, distilling further. As for after desiccation, it is desirable to save under an inert gas ambient atmosphere with a moisture content of 100 ppm or less.
[0014]
The moisture content of at least a kind of solvent in which purification processing was carried out by dehydration part processing and/or column purification has desirable 100 ppm or less, its 50 ppm or less are more desirable, and its 30

ppm or less are still more desirable. The impurities which degrade moisture and/or the luminescence engine performance of a light emitting device are reducible with dehydration part processing and/or column purification of a solvent.

[0015]
In column purification, especially the bulking agent used for a column is not limited, for example, can use suitably silica gel, an alumina, cationic ion exchange resin, anionic ion exchange resin, etc. Moreover, basicity and any neutral and acid bulking agent can be used.

[0016]
As for the amount of a bulking agent, it is desirable that it is 50g or more 1kg or less to 100ml of solvents to refine. If fewer than this, the effectiveness of purification will become small, if [than this] more, the amount of a bulking agent will become useless and productive efficiency will fall. Moreover, as for the flow rate at the time of purification, it is desirable that it is [1ml] the following by 1000ml/above by /. If there are few flow rates than this, productive efficiency will worsen. Moreover, the effectiveness of purification is not small desirable if [than this] more.

[0017]
By carrying out column purification of the chlorine-based solvent, it is surmised that chlorine active species, such as a radical and a cation, are removed. If especially chlorine active species remains in a solvent, the ingredient contained in the organic layer mentioned later will be chlorinated, and it will become the cause of endurance aggravation and effectiveness aggravation.

[0018]
In the manufacture approach of this invention, especially a desirable mode is formation of the luminous layer using the coating liquid containing at least a kind of solvent by which purification processing was carried out, and at least one phosphorescence luminescent material. Purification processing has desirable column purification and its concomitant use with column purification and dehydration part processing is more desirable. An alt.metal-ized metal complex etc. is preferably mentioned so that a phosphorescence luminescence ingredient may be explained in full detail below. As for a solvent, it is desirable that it is the solvent which contains a kind of halogen system solvent at least.

[0019]
especially desirable voice -- as for the solvent which can be set like and by which purification processing was carried out, it is desirable that it is the solvent from which impurities, such as chlorine active species, such as a radical and a cation, were removed below to a certain concentration. As for the concentration of the alt.metal-ized metal complex chlorinated in the luminous layer, it is desirable that it is below a certain concentration.

[0020]
(Light emitting device)
The light emitting device in this invention is stated to a detail below. It is the layer which the organic layer of this invention means the layer which can be used for a light emitting device, and an organic layer mainly consists of an organic compound, and specifically contains the compound which has the operation effectiveness as a luminescent organic layer, an electronic transportability organic layer, a hole transportability organic layer, an electronic injection layer, a hole impregnation layer, etc. In addition, in the following, the vocabulary a "derivative" means the compound itself and its derivative, for example, a coumarin derivative means a coumarin and its derivative.

[0021]
-- Configuration of an organic layer --
Although there is especially no limit and it can choose suitably according to the application and the object of a light emitting device as a formation location in said light emitting device of said organic layer, it is desirable to be formed between the first electrode and the second electrode. Either a transparent electrode or a back plate is OK as said first electrode and the second electrode. An organic layer is formed in the front face or the whole surface on said transparent electrode or said back plate. About the configuration of said organic layer, magnitude, and thickness, there is especially no limit and it can be suitably chosen according to the object.

[0022]
As concrete lamination, a transparence anode plate / luminous layer / cathode, a transparence anode plate / luminous layer / electronic transporting bed / cathode, a transparence anode plate / electron hole transporting bed / luminous layer / electronic transporting bed / cathode, a transparence anode plate / electron hole transporting bed / luminous layer / cathode, a transparence anode plate / luminous layer / electronic transporting bed / electronic injection layer / cathode, a transparence anode plate / hole injection layer / electron hole transporting bed / luminous layer / electronic transporting bed / electronic injection layer / cathode, etc. are mentioned.

[0023]
-- Luminous layer --
The luminous layer used for this invention may consist of a kind of luminescence material at least, and may also contain electron hole transport material, electronic transport material, and host material if needed. It is not limited especially as luminescence material used for this invention, and if it is a fluorescence luminescence compound or a phosphorescence luminescence compound, it can use.

[0024]
As a fluorescence luminescence compound, for example, a benzooxazole derivative, a benzimidazole derivative, A benzothiazole derivative, a styryl benzene derivative, a polyphenyl derivative, A diphenyl butadiene derivative, a tetra-phenyl butadiene derivative, the North America Free Trade Agreement RUIMIDO derivative, A coumarin derivative, a perylene derivative, a peri non derivative, an OKISA diazo-RU derivative, An aldazine derivative, a PIRARIJIN derivative, a cyclopentadiene derivative, A bis-styryl anthracene derivative, the Quinacridone derivative, a pyrrolo pyridine derivative, A thiadiazolo pyridine derivative, a styryl amine derivative, an aromatic series dimethylidene compound, High molecular compounds, such as various metal complexes represented by the metal complex and rare earth complex of an eight-quinolinol derivative, the poly thiophene derivative, a polyphenylene derivative, a polyphenylene vinylene derivative, and the poly fluorene derivative, etc. are mentioned. These can mix and use a kind or two sorts or more.

[0025]
Although not limited especially as a phosphorescence luminescence compound, an alt.metal-ized metal complex or a porphyrin metal complex is desirable.

[0026]

Said alt.metal-ized metal complex is the generic name of the compound group indicated by Akio Yamamoto work "organic metal chemistry-foundation and application" 150 page, 232 pages, Shokabo Publishing Co., Ltd. (1982 issuance), the "Photochemistry and Photophisics of Coodination Compounds" 71-77 page written by H.Yersin, 135-146 pages and Springer-Verlag (1987 issuance), etc. [-] Said organic layer containing this alt.metal-ized metal complex is advantageous at the point of excelling in luminous efficiency by high brightness.

[0027]

As a ligand which forms an alt.metal-ized metal complex, there are various things, and it is indicated by the above-mentioned reference, and 2-phenyl pyridine derivative, 7, 8-benzoquinoline derivative, 2-(2-thienyl) pyridine derivative, 2-(1-naphthyl) pyridine derivative, 2-phenyl quinoline derivative, etc. are mentioned as a desirable ligand also in it. These derivatives may have a substituent if needed. The alt.metal-ized metal complex may have other ligands other than said ligand.

[0028]

alt.metal-ized metal complex used by this invention Inorg.Chem. 1991 and No. 30, 1685page., ** 1988 and No. 27, 3464page., ** 1994 and No. 33, 545 page .Inorg.Chim.Acta 1991, No. 181, 245 page .J.Organomet.Chem. 1987, No. 335, and 293page.J.Am.Chem.Soc. 1985, No. 107, and 1431page. etc. — It is compoundable by various well-known technique.

[0029]

Also in said alt.metal-ized complex, the compound which emits light from a triplet exciton can use it suitably from a viewpoint of the improvement in luminous efficiency in this invention. Moreover, in a porphyrin metal complex, a porphyrin platinum complex is desirable.

[0030]

The compound of said phosphorescence luminescence may be used by the one-sort independent, and may use two or more sorts together. Moreover, said fluorescence luminescence compound and a phosphorescence luminescence compound may be used simultaneously. In this invention, it is desirable to use said phosphorescence luminescence compound from the point of luminescence brightness and luminous efficiency.

[0031]

As a content in said luminous layer of the compound of said phosphorescence luminescence, there is especially no limit, and it can be suitably chosen according to the object, for example, is 0.1 - 70 % of the weight, and its 1 - 20 % of the weight is desirable. When the content of the compound of said phosphorescence luminescence is not 0.1 - 70 % of the weight, the content effectiveness is enough in the content effectiveness fully not being demonstrated and it being 1 - 20 % of the weight.

[0032]

Said host compound is a compound which has the function for energy transfer to arise from the excitation state to the compound of said phosphorescence luminescence, consequently to make the compound of this phosphorescence luminescence emit light.

[0033]

If it is the compound which can carry out energy transfer of the exciton energy to luminescence material as said host material, there will be especially no limit. According to the object, it can choose suitably. Specifically A carbazole derivative, A triazole derivative, an oxazole derivative, an OKISA diazo-RU derivative, An imidazole derivative, the poly aryl alkane derivative, a pyrazoline derivative, A pyrazolone derivative, a phenylenediamine derivative, an arylamine derivative, an amino permutation chalcone derivative, a styryl anthracene derivative, and full — me — non — a derivative — A hydrazone derivative, a stilbene derivative, a silazane derivative, an aromatic series tertiary-amine compound, A styryl amine compound, an aromatic series dimethylidene system compound, a porphyrin system compound, An anthra quinodimethan derivative, an anthrone derivative, a diphenyl quinone derivative, A thiopyran dioxide derivative, a carbodiimide derivative, a full ORENIRIDEN methane derivative, Heterocycle tetracarboxylic acid anhydrides, such as a JISUCHIRIRU pyrazine derivative and naphthalene perylene, The metal complex and metal phthalocyanine of a phthalocyanine derivative and an eight-quinolinol derivative, The various metal complex polysilane system compounds represented by the metal complex which makes benzooxazole and benzothiazole a ligand, The Pori (N-vinylcarbazole) derivative, an aniline system copolymer, thiophene oligomer, High molecular compounds, such as conductive polymer oligomer, such as the poly thiophene, the poly thiophene derivative, a polyphenylene derivative, a polyphenylene vinylene derivative, and the poly fluorene derivative, etc. are mentioned. Said host compound may be used by the one-sort independent, and may use two or more sorts together.

[0034]

Furthermore, in this invention, electron hole transport material and electronic transport material may be included in said luminous layer if needed.

[0035]

as said electron hole transport material — low-molecular electron hole transport material and macromolecule electron hole transport material — if all can be used, it has the function to pour in an electron hole from an anode plate, the function to convey an electron hole, or the function that carries out the obstruction of the electron poured in from cathode and it is, it will not be limited, for example, the following ingredients can be mentioned.

[0036]

A carbazole derivative, a triazole derivative, an oxazole derivative, an OKISA diazo-RU derivative, An imidazole derivative, the poly aryl alkane derivative, a pyrazoline derivative, A pyrazolone derivative, a phenylenediamine derivative, an arylamine derivative, an amino permutation chalcone derivative, a styryl anthracene derivative, and full — me — non — a derivative — A hydrazone derivative, a stilbene derivative, a silazane derivative, an aromatic series tertiary-amine compound, A styryl amine compound, an aromatic series dimethylidene system compound, a porphyrin system compound, A polysilane system compound, the Pori (N-vinylcarbazole) derivative, an aniline system copolymer, High molecular compounds, such as conductive polymer oligomer, such as thiophene oligomer and the poly thiophene, the poly thiophene derivative, a polyphenylene derivative, a polyphenylene vinylene derivative, and the poly fluorene derivative, etc. are mentioned. These may be used by the one-sort independent and may use two or more sorts together.

[0037]

If it has the function to convey an electron as said electronic transport material, or the function which carries out the obstruction of the electron hole poured in from the anode plate and is, it will not be restricted, for example, the

following ingredients can be mentioned. A triazole derivative, an oxazole derivative, an OKISA diazo-RU derivative, full — me — non — a derivative, an anthra quinodimethan derivative, and an anthrone derivative — A diphenyl quinone derivative, a thiopyran dioxide derivative, a carbodiimide derivative, Heterocycle tetracarboxylic acid anhydrides, such as a full ORENIRIDEN methane derivative, a JISUCHIRIRU pyrazine derivative, and naphthalene perylene, The metal complex and metal phthalocyanine of a phthalocyanine derivative and an eight-quinolinol derivative, The various metal complexes represented by the metal complex which makes benzooxazole and benzothiazole a ligand, High molecular compounds, such as conductive polymer oligomer, such as an aniline system copolymer, thiophene oligomer, and the poly thiophene, the poly thiophene derivative, a polyphenylene derivative, a polyphenylene vinylene derivative, and the poly fluorene derivative, can be mentioned.
Furthermore in this invention, an inactive polymer-binder can be electrically used for a luminous layer if needed.
[0038]
As an inactive polymer-binder, a polyvinyl chloride, a polycarbonate, polystyrene, polymethylmethacrylate, poly butyl methacrylate, polyester, polysulfone, polyphenylene oxide, polybutadiene, hydrocarbon resin, ketone resin, phenoxy resin, a polyamide, ethyl cellulose, vinyl acetate, ABS plastics, polyurethane, melamine resin, unsaturated polyester, alkyd resin, an epoxy resin, silicon resin, a polyvinyl butyral, a polyvinyl acetal, etc. can be mentioned to the electric target used if needed, for example. If said luminous layer contains said polymer-binder, it is advantageous at the point which can carry out spreading formation of this luminous layer easily by the wet producing-film method at a large area.
[0039]
As thickness of said luminous layer, 10-200nm is desirable and 20-80nm is more desirable. When said thickness exceeds 200nm, driver voltage may go up, and when it is less than 10nm, this light emitting device may short-circuit.
[0040]
— Electronic transporting bed —
The electronic transporting bed which contains electronic transport material if needed in this invention can be prepared. It will not be restricted, if it has the function to convey an electron as said electronic transport material, or the function which carries out the obstruction of the electron hole poured in from the anode plate and is, and said electronic transport material can be used suitably.
[0041]
Moreover, an inactive polymer-binder can be electrically used for said electronic transporting bed if needed, and said polymer-binder can be used for it. As thickness of said electronic transporting bed, 10-200nm is desirable and 20-80nm is more desirable. When said thickness exceeds 200nm, driver voltage may go up, and when it is less than 10nm, this light emitting device may short-circuit.
[0042]
— Electron hole transporting bed —
The electron hole transporting bed which contains electron hole transport material if needed in this invention can be prepared. It will not be restricted, if it has the function to convey an electron hole as said electron hole transport material, or the function which carries out the obstruction of the electron poured in from cathode and is, and said electron hole transport material can be used suitably.
[0043]
Moreover, an inactive polymer-binder can be electrically used for said electron hole transporting bed if needed, and said polymer-binder can be used for it. As thickness of said electron hole transporting bed, 10-200nm is desirable and 20-80nm is more desirable. When said thickness exceeds 200nm, driver voltage may go up, and when it is less than 10nm, this light emitting device may short-circuit.
[0044]
— Formation of an organic layer —
In this invention, much more organic layer at least is formed by the wet producing-film method. other organic layers — the dry type producing-film method and the wet producing-film method — a film may be produced by which approach. Vacuum deposition, a spatter, etc. are mentioned as a dry type producing-film method. As a wet producing-film method, those, such as dipping, a spin coat method, a dip coating method, the cast method, the DAIKO-TO method, the roll coat method, the bar coat method, and the GURABIAKO-TO method, is mentioned, and a film can be suitably produced by all.
The light emitting device which could large-area-ize said organic layer easily, and was excellent in luminous efficiency especially with high brightness in the spreading formation by said wet producing-film method is advantageous at the point efficiently acquired by low cost. In addition, selection of the class of these producing-film methods can be suitably performed according to the ingredient of this organic layer. Moreover, when carrying out a laminating more than two-layer by the wet producing-film method, in order to avoid mixing with a multilayer, a replica method and print processes can also be used suitably.
[0045]
Although it can dry suitably and there is especially no limit as conditions for this desiccation after producing a film when a film is produced by said wet producing-film method, the temperature of the range which the layer which carried out spreading formation does not damage etc. is employable.
[0046]
In addition, as an amount of solid content to the amount solvent of solid content in said coating liquid, there is especially no limit and the viscosity can also choose it as arbitration according to the wet film production approach.
[0047]
- Base material -
In this invention, as an ingredient used as a base material Specifically For example, inorganic materials, such as YSZ (zirconia stabilization yttrium) and glass, Polyester, such as polyethylene terephthalate, polybutylene terephthalate, and polyethylenenaphthalate, Polystyrene, a polycarbonate, polyether sulphone, polyarylate, Polymeric materials, such as allyl compound diethylene glycol carbonate, polyimide, the poly cycloolefin, norbornene resin, Pori (chlorotrifluoroethylene), Teflon, and a polytetrafluoroethylene-polyethylene copolymer, etc. are mentioned. Especially, in the case of a flexible light emitting device and a spreading mold light emitting device, it is desirable that they are polymeric materials.
[0048]
Even inside of polymeric materials, It is desirable that they are the polymeric materials which contain fluorine atoms,

such as polyester, a polycarbonate, polyether sulphone or Pori (chlorotrifluoroethylene), Teflon, and a polytetrafluoroethylene-polyethylene copolymer, from viewpoints, such as oxygen permeability, transparency, thermal resistance, dimensional stability, solvent resistance, electric insulation, workability, low permeability, and low hygroscopicity.

[0049]

About the configuration of said base material, structure, and magnitude, there is especially no limit and it can be suitably chosen according to the application of a light emitting device, the object, etc. Generally, as said configuration, it is tabular. As said structure, you may be monolayer structure, and may be a laminated structure, and it may be formed by the single member, and may be formed by two or more members.

[0050]

It is desirable that said base material may be transparent and colorless, and it is transparent and colorless at the point to which dispersion or attenuation does not carry out light emitted from said luminous layer although you may be colored transparence.

[0051]

A moisture permeation prevention layer (gas barrier layer) can be prepared in the front face or rear face (said transparent electrode side) at said base material. As an ingredient of said moisture permeation prevention layer (gas barrier layer), inorganic substances, such as silicon nitride and oxidation silicon, are used suitably. This moisture permeation prevention layer (gas barrier layer) can be formed for example, by a RF-sputtering method etc. A rebound ace court layer, an under coat layer, etc. may be further prepared in said base material if needed.

[0052]

- Transparent electrode -

That what is necessary is just to usually have the function as an anode plate which supplies an electron hole to said organic layer as said transparent electrode, there is especially no limit about the configuration, structure, and magnitude, and it can choose suitably from well-known electrodes according to the application of a light emitting device, and the object. What is necessary is also being able to operate said transparent electrode as cathode and making it just operate said back plate as an anode plate in this case.

[0053]

As an ingredient of said transparent electrode, for example, a metal, an alloy, a metallic oxide, organic conductivity compounds, or such mixture can be mentioned suitably, and an ingredient 4.0eV or more has a desirable work function. the tin oxide (ATO —) which doped antimony, a fluorine, etc. as an example FTO, tin oxide, a zinc oxide, indium oxide, indium oxide tin (ITO), Semiconductance metallic oxides, such as a zinc oxide indium (IZO), gold, silver, chromium, Organic conductivity ingredients, such as inorganic conductivity matter, such as mixture of these metals and conductive metallic oxide or laminated material, copper iodide, and copper sulfide, the poly aniline, the poly thiophene, and polypyrrole, the laminated material of these and ITO, etc. are mentioned to metals, such as nickel, and a pan.

[0054]

Said transparent electrode can be formed on said substrate according to the approach suitably chosen in consideration of fitness with said ingredient from chemical methods, such as physical methods, such as wet methods, such as a printing method and a coating method, vacuum evaporation technique, the sputtering method, and the ion plating method, CVD, and a plasma-CVD method, etc. For example, as an ingredient of said transparent electrode, when choosing ITO, formation of this transparent electrode can be performed according to a direct current or a high frequency spatter, vacuum evaporation technique, the ion plating method, etc. Moreover, when choosing an organic conductivity compound as an ingredient of said transparent electrode, it can carry out according to the wet producing-film method.

[0055]

Although there is especially no limit and it can choose suitably according to the application of this light emitting device, and the object as a formation location in said light emitting device of said transparent electrode, being formed on said substrate is desirable. in this case, also in said substrate, a front face boils all, it may be formed and this transparent electrode may be formed in that part.

[0056]

In addition, the chemical etching by photolithography etc. may perform patterning of said transparent electrode, physical etching by laser etc. may perform it, and it may carry out vacuum deposition, a spatter, etc. in piles, may perform a mask, and may perform it by the lift-off method or print processes.

[0057]

Although it can choose suitably with said ingredient and cannot generally specify as thickness of said transparent electrode, it is usually 10nm - 50 micrometers, and 50nm - 20 micrometers are desirable. As resistance of said transparent electrode, below 103ohms / ** are desirable, and below 102ohms / ** are more desirable. It may be transparent and colorless, or may be colored transparence, and in order to take out luminescence from this transparent electrode side, as the permeability, said 60% or more of transparent electrode is desirable, and is more desirable. [70% or more of] This permeability can be measured according to the well-known approach which used the spectrophotometer.

[0058]

In addition, about said transparent electrode, the Sawada ****** "new expansion of transparent electrode film" CMC ** (1999) has a detailed description, and these can be applied to this invention. When using a heat-resistant low plastic base material, the transparent electrode which used ITO or IZO and produced the film at low temperature 150 degrees C or less is desirable.

[0059]

-- Back plate --

That what is necessary is just to usually have the function as cathode to inject an electron into said organic layer, as said back plate, there is especially no limit about the configuration, structure, and magnitude, and it can choose suitably from well-known electrodes according to the application of a light emitting device, and the object.

What is necessary is also being able to operate said back plate as an anode plate, and making it just operate said transparent electrode as cathode in this case.

[0060]

As an ingredient of said back plate, for example, a metal, an alloy, a metallic oxide, electrical conductivity compounds,

such mixture, etc. are mentioned, and a thing 4.5eV or less has a desirable work function. As an example, rare earth metals, such as alkali metal (for example, Li, Na, K, Cs, etc.), alkaline earth metal (for example, Mg, calcium, etc.), gold, silver, lead, aluminum, a sodium-potassium alloy, a lithium-aluminium alloy, a magnesium-silver alloy, an indium, and an ytterbium, etc. are mentioned. Although these may be used by the one-sort independent, they can use two or more sorts together suitably from a viewpoint which reconciles stability and electron injection nature.

[0061]

Also in these, in respect of electron injection nature, alkali metal and an alkalinity metal are desirable and the ingredient which makes aluminum a subject in that it excels in preservation stability is desirable.

The ingredient which makes said aluminum a subject means an alloy or mixture with aluminum independence or aluminum, 0.01 - 10% of the weight of alkali metal, or an alkaline earth metal (for example, a lithium-aluminum alloy, a magnesium-aluminum alloy, etc.).

[0062]

In addition, the ingredient of said back plate is explained by JP,2-15595,A and JP,5-121172,A in full detail.

[0063]

There is no method of forming said back plate, and it can perform especially a limit according to a well-known approach. For example, according to the approach suitably chosen in consideration of fitness with said ingredient from chemical methods, such as physical methods, such as wet methods, such as a printing method and a coating method, vacuum evaporation technique, the sputtering method, and the ion plating method, CVD, and a plasma-CVD method, etc., it can form on said substrate. For example, as an ingredient of said back plate, when choosing a metal etc., its one sort or two sorts or more can be performed to coincidence or sequential according to a spatter etc.

[0064]

In addition, the chemical etching by photolithography etc. may perform patterning of said back plate, physical etching by laser etc. may perform it, and it may carry out vacuum deposition, a spatter, etc. in piles, may perform a mask, and may perform it by the lift-off method or print processes.

[0065]

Although there is especially no limit and it can choose suitably according to the application of this light emitting device, and the object as a formation location in said light emitting device of said back plate, being formed on said organic layer is desirable. In this case, this back plate may be formed in all on said organic layer, and may be formed in that part. Moreover, the dielectric layer by the fluoride of said alkali metal or said alkaline earth metal etc. may be inserted by the thickness of 0.1-5nm between said back plates and said organic layers. In addition, this dielectric layer can be formed by vacuum evaporation technique, the sputtering method, the ion plating method, etc.

[0066]

Although it can choose suitably with said ingredient and cannot generally specify as thickness of said transparent electrode, it is usually 10nm - 5 micrometers, and 50nm - 1 micrometer is desirable. Said back plate may be transparent and may be opaque. In addition, a transparent back plate can produce the ingredient of said back plate thinly in thickness of 1-10nm, and can form it by carrying out the laminating of the conductive ingredient with said still more transparent ITO, IZO, etc.

[0067]

- Other layers -

As a layer of said others, there is especially no limit, and it can be suitably chosen according to the object, for example, a protective layer etc. is mentioned. As said protective layer, the thing of a publication is suitably mentioned to JP,7-85974,A, a 7-192866 official report, a 8-22891 official report, a 10-275682 official report, a 10-106746 official report, etc., for example.

[0068]

In said layered product component, said protective layer is formed on this back plate, when the laminating of said base material, said transparent electrode, said organic layer, and said back plate is carried out to this order, and when the laminating of said base material, said back plate, said organic layer, and said transparent electrode is carried out to this order, it is formed on this transparent electrode in that outermost surface, for example.

[0069]

About the configuration of said protective layer, magnitude, and thickness, it can choose suitably, as the ingredient, if it has the function which controls making what may degrade light emitting devices, such as moisture and oxygen, invade thru/or penetrate in this light emitting device, there will be especially no limit, for example, oxidation silicon, a silicon dioxide, a germanium dioxide, diacid-ized germanium, etc. are mentioned.

[0070]

As the formation approach of said protective layer, there is especially no definition, for example, a vacuum deposition method, the sputtering method, a reactive sputtering method, the molecule SENEPI taxi method, the ionized cluster beam method, the ion plating method, a plasma polymerization method, a plasma-CVD method, a laser CVD method, a heat CVD method, a coating method, etc. are mentioned.

[0071]

Furthermore, in this invention, it is the object which prevents the trespass of the moisture to each class, or oxygen in said light emitting device, and it is also desirable to prepare a closure layer. The copolymer which contains tetrafluoroethylene and at least one sort of comonomers as an ingredient of said closure layer, for example, The fluorine-containing copolymer which has cyclic structure in a copolymerization principal chain, polyethylene, polypropylene, Polymethylmethacrylate, polyimide, polyurea, polytetrafluoroethylene, Polychlorotrifluoroethylene resin, poly dichlorodifluoroethene, Two or more sorts of copolymers chosen from chlorotrifluoroethylene and dichlorodifluoroethene, The absorptivity matter of 1% or more of water absorption, the dampproof matter of 0.1% or less of water absorption, In, Metals, such as Sn, Pb, Au, Cu, Ag, aluminum, Tl, and nickel, MgO, SiO, SiO2, aluminum 2O3, GeO, NiO, CaO, BaO, The metallic oxide of Fe 2O3, Y2O3, and TiO2 grade, MgF2, LiF, The thing which made liquefied fluorination carbon, such as AlF3, a metal fluoride of CaF2 grade, par fluoroalkane, a perfluoro amine, and par full OROE-Tell, and liquefied fluorination carbon distribute the adsorbent which adsorbs moisture and oxygen is mentioned.

[0072]

Moreover, in this invention, it is desirable to close said light emitting device with a closure plate and a closure container using encapsulant for moisture with the exterior or the object of cutoff of oxygen. As construction material used for a closure plate and a closure container, plastics, ceramics, etc., such as metals, such as glass, stainless steel, and

aluminum, Pori (chlorotrifluoroethylene), polyester, and a polycarbonate, can be used. as a sealing agent — ultraviolet-rays hardening resin, heat-curing resin, and 2 liquid mold-curing resin — all can be used.

[0073]

In this invention, a water absorption agent or an inactive liquid can be installed in the space between a closure container and a light emitting device. although not limited especially as a water absorption agent — for example, barium-oxide, sodium oxide, potassium oxide, calcium-oxide, sodium-sulfate, calcium-sulfate, magnesium sulfate, phosphorus pentoxide, calcium chloride, magnesium chloride, copper chloride, cesium fluoride, and niobium fluoride, a calcium bromide, and bromination — vanadium, a molecular sieve, a zeolite, a magnesium oxide, etc. can be mentioned. Especially as an inactive liquid, although not limited, fluorine system solvents, such as paraffin, liquid paraffins, par fluoroalkane, and a perfluoro amine, par full OROE-Tell, a chlorine-based solvent, and silicone oil are mentioned.

[0074]

The light emitting device of this invention can obtain luminescence by impressing a direct-current (alternating current component also being included if needed) electrical potential difference (usually 2 volts - 40 volts) or a direct current between said transparent electrodes and said back plates.

[0075]

The approach of a publication can be used for JP,2-148687,A, 6-301355, 5-29080, 7-134558, 8-234685, said 241047 numbers, a U.S. Pat. No. 5828429 number, said 6023308 numbers, the Japanese patent No. 2784615, etc. about actuation of the light emitting device of this invention.

[0076]

[Translation done.]

EXAMPLE

[Example]
Although the example of the light emitting device of this invention is explained below, this invention is not limited at all by these examples. As long as it was unstated, preparation of the coating liquid of an example and formation of an organic layer were performed in the nitrogen-purge glove compartment with a moisture of 50 ppm or less.
[0077]
(Example 1-1)
As a base material, thickness cut 0.2mm glass plate on 2.5cm square, and introduced in the vacuum chamber, and SnO2 content formed the ITO thin film (thickness of 0.2 micrometers) as a transparent electrode using the ITO target which is 10 % of the weight by DC magnetron sputtering (conditions: base material temperature of 100 degrees C, oxygen pressure 1x10-3Pa). The surface electrical resistance of an ITO thin film was 10ohm/**.
[0078]
Next, the substrate in which said transparent electrode was formed was put into the wash fountain, and after carrying out IPA washing, UV-ozonization was performed to this for 30 minutes. And it is SUPINKO to the front face of this transparent electrode about Pori (ethylene dioxythiophene) and a polystyrene sulfonate water distribution object (the product made from BAYER, Baytron P: 1.3% of solid content). - After carrying out TO, the vacuum drying was carried out for 2 hours, and 150 degrees C of hole injection layers whose thickness is 100nm were formed.
[0079]
100g (200 meshes, product made from the Wako Pure Chem industry) of activated aluminas was put into glass column tubing with a diameter [of 3cm], and a die length of 50cm, and it refined by on the other hand passing 1,2-dichloroethane (object for high-speed liquid chromatographs, product made from Wako Pure Chem industry) 50ml which is the solvent of luminous layer coating liquid by part for 10ml/of the rates of flow using this column.
[0080]
2 hours after performing the above-mentioned purification processing of 1,2-dichloroethane As an alt.metal-ized complex which is the polyvinyl carbazole (Mw=63000, Aldrich make) and phosphorescence luminescence material as host [electron hole transport material-cum-] material 1, 3, and 4-OKISA diazo-RU (PBD) is dissolved in the above-mentioned purification dichloroethane by the weight ratio of 40:1:12. 2-(4-biphenylyl)-5-(4-t-buthylphenyl)- which is a ** tris (2-phenyl pyridine) iridium complex and electronic transport material -- Luminous layer coating liquid was prepared.
[0081]
This coating liquid was applied on said hole injection layer using the spin coater, and thickness formed the luminous layer which is 100nm by making it dry at a room temperature.
Furthermore, the mask (mask with which luminescence area is set to 5mmx5mm) which carried out patterning was installed on this luminous layer, magnesium:silver =10:1 [0.25-micrometer] (mole ratio) was vapor-deposited within vacuum evaporationo equipment, 0.3 micrometers of silver were vapor-deposited, and the back plate was formed. From said transparent electrode (it functions as an anode plate) and said back plate, the lead wire of aluminum was connected, respectively and the laminating structure was formed.
[0082]
The laminating structure obtained here was put in in the glove compartment permuted with nitrogen gas, and was closed using ultraviolet curing mold adhesives (the product made from the Nagase tiba, XNR5493) with the glass closure container. The light emitting device of an example 1 was created by the above.
[0083]
(Example 1-2)
In the example 1-1, except using chloroform/methyl ethyl ketone (70/30 volume %) instead of 1 and 2-dichloroethane, it refined by the same approach as an example 1-1, and the light emitting device was produced and the solvent of a luminous layer was evaluated.
[0084]
(Example 1-3)
In the example 1-1, instead of carrying out column purification of the 1,2-dichloroethane which is the solvent of a luminous layer, and carrying out coating liquid adjustment 2 hours after, it is the same approach as an example 1-1, and the light emitting device was produced and evaluated except carrying out coating liquid preparation three days after.
[0085]
(Example 1-4)
In the example 1-1, instead of carrying out column purification of the 1,2-dichloroethane which is the solvent of a luminous layer, and carrying out coating liquid adjustment 2 hours after, it is the same approach as an example 1-1, and the light emitting device was produced and evaluated except carrying out coating liquid preparation seven days after.
[0086]
(Example 1-1 of a comparison)
In the example 1-1, except not carrying out column purification of the 1,2-dichloroethane which is the solvent of a luminous layer, it is the same approach as an example 1-1, and the light emitting device was produced and evaluated.
[0087]
(The assessment approach of a light emitting device)
Direct current voltage was impressed to the organic EL device, and the light emitting device in the above-mentioned

example and the example of a comparison was made to emit light using the source major unit 2400 mold made from Oriental TEKUNIKA. The electrical potential difference when Lmax and Lmax are obtained in the highest brightness at that time was set to Vmax. Moreover, the luminous efficiency at 200 Cd/m 2:00 (eta 200) was shown in a table 1. Furthermore, constant current continuation actuation of this component was carried out by initial brightness 200 Cd/m2, time amount (half-life) from which brightness became half was set to T1/2, and it was shown in a table 1.
[0088]
[A table 1]

	L_{max} (cd／m²)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例1-1	88000	15	13.2	12000
実施例1-2	78000	15	12.3	11000
実施例1-3	69000	15	12.1	10000
実施例1-4	65000	15	11.4	9500
比較例1-1	42000	17	8.1	4800

[0089]
It turns out that a solvent is not refined in the coating liquid in the example 1-1 of a comparison, but luminescence brightness, luminous efficiency, and endurance are inferior to the table compared with examples 1, 2, 3, and 4.
[0090]
(Example 2-1)
The transparent electrode (ITO thin film) and the base material which has a hole injection layer were created like the example 1-1.
[0091]
1,2-dichloroethane (object for high-speed liquid chromatographs, product made from Wako Pure Chem industry) 300ml which is the solvent of luminous layer coating liquid, and 100g of anhydrous sodium sulfate were put into the 500ml mold flask to make, and it was left one whole day and night, and dried. It filtered after desiccation, it distilled further and dehydration processing (moisture content of 30 ppm) was performed. The 1,2-dichloroethane which carried out dehydration processing was saved in the nitrogen gas globe box with a moisture content of 50 ppm.
[0092]
2 hours after performing the above-mentioned dehydration processing of 1,2-dichloroethane As an alt.metal-ized complex which is the polyvinyl carbazole (Mw=63000, Aldrich make) and phosphorescence luminescence material as host [electron hole transport material-cum-] material 2-(4-biphenylyl)-5-(4-t-buthylphenyl)- which is a ** tris (2-phenyl pyridine) iridium complex and electronic transport material — 1, 3, and 4-OKISA diazo-RU (PBD) to the above-mentioned dehydration 1,2-dichloroethane by the weight ratio of 40:1:12 It dissolved in the nitrogen gas globe box with a moisture content of 50 ppm, and luminous layer coating liquid was prepared.
[0093]
Thickness formed the luminous layer which is 100nm by applying this coating liquid on said hole injection layer using a spin coater in a nitrogen gas globe box with a moisture content of 50 ppm, and drying it at a room temperature.
[0094]
Like the example 1-1, the back plate was formed on the luminous layer, from said transparent electrode (it functions as an anode plate) and said back plate, the lead wire of aluminum was connected, respectively and the laminating structure was formed.
[0095]
The light emitting device was created like the example 1-1 except closing the laminating structure obtained here in a nitrogen gas globe box with a moisture content of 50 ppm. The evaluation criteria of the light emitting device in the following table are the same as it of an example 1-1.
[0096]
(Example 2-2)
In the example 2-1, except using chloroform/methyl ethyl ketone (70/30 volume %; moisture content of 50 ppm) instead of 1 and 2-dichloroethane, it refined by the same approach as an example 2-1, and the light emitting device was produced and the solvent of a luminous layer was evaluated.
[0097]
(Example 2-3)
In the example 2-1, instead of carrying out dehydration processing of the 1,2-dichloroethane which is the solvent of a luminous layer, and carrying out coating liquid adjustment 2 hours after, it is the same approach as an example 2-1, and the light emitting device was produced and evaluated except carrying out coating liquid preparation seven days after. The moisture content of the coating liquid of seven days after was 90 ppm.
[0098]

(Example 2-1 of a comparison)
In the example 2-1, except not carrying out dehydration processing of the 1,2-dichloroethane which is the solvent of a luminous layer, it is the same approach as an example 2-1, and the light emitting device was produced and evaluated. In addition, the moisture content of the 1,2-dichloroethane which does not carry out dehydration processing was 800 ppm.
[0099]
Assessment of the above-mentioned light emitting device was performed like the above-mentioned example (1-1).
[0100]
[A table 2]

	L_{max} (cd／m²)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例2-1	78000	15	12.2	11000
実施例2-2	72000	15	12.3	12000
実施例2-3	59000	15	11.0	9000
比較例2-1	40000	17	7.9	4200

[0101]
With the coating liquid in the example 2-1 of a comparison, dehydration processing of the solvent is not carried out, but luminescence brightness, luminous efficiency, and endurance are inferior to the above-mentioned table compared with an example 2-1, 2-2, and 2-3.
[0102]
(Example 3-1)
Like the example 1-1, the ITO thin film was formed on the glass substrate, and then the hole injection layer was formed. On the other hand, like the example 1-1, column purification of the 1,2-dichloroethane was carried out, dehydration processing of this 1,2-dichloroethane was further carried out like the example 2-1, and 1,2-dichloroethane with a moisture content of 10 ppm was obtained. the refined 1,2-dichloroethane -- using -- an example 2-1 -- the same -- carrying out -- luminous layer coating liquid -- preparation, luminous layer formation, and a laminating -- it closed and the light emitting device was created. The evaluation criteria in the following table are the same as an example 1.
[0103]
[A table 3]

	L_{max} (cd／m²)	V_{max} (V)	η_{200} (%)	$T_{1/2}$ (時間)
実施例3-1	96000	14	15.3	14000

[0104]
It turns out that the case of purification processing that the combination with dehydration processing of a solvent and column purification is more independent than a table is excelled in high luminescence brightness, low-battery actuation, high luminous efficiency, and endurance.
[0105]

[Translation done.]